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CIVIL ENGINEERING LABORATORY
Naval Construction Battalion Center
Port Hueneme, California

10 Theo J. /West

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FABRICATION OF EIBERGLASS REINFORCED PLASTIC SURFACING UNDER WET CONDITIONS

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It was established that the resin viscosity should be less than 100 cps at the temperature of application for optimum flexural strength. The incorporation of a surfactant into the wet fiberglass mat was found to result in improved resin penetration into the woven roving and increased flexural strength. The addition of 15 percent styrene to the presently used Port Hueneme resin (Selection RS50338) and the incorporation of Triton X100 into the water in the fiberglass mat results in flexural strengths of 25,000 psi - a considerable improvement over the 4,000 psi obtained with undiluted Port Hueneme resin.

DERAKANE\* 510-A-40 when diluted with 15 percent styrene and, used with Triton X100, gives laminates having flexural strengths of 30,000 psi, meeting the 28,000 psi flexural strength specification. This resin does not have the desired five year shelf life.

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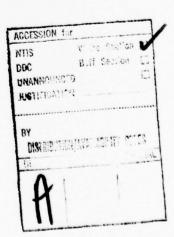
#### **ABSTRACT**

The purpose of this investigation was to develop a system to be used in the construction of fiberglass-reinforced plastic surfacings for soil under wet conditions. The effect of water on laminate properties, in the substrate and in the fiberglass mat has been defined and solutions have been developed for some of the problems. A system of chemical components has been developed for the field placement of the reinforced plastic soil surfacings under wet conditions, that is superior to that presently used and is usable with the basic spray equipment now used by the Marine Corps.

It was established that the resin viscosity should be less than 100 cps at the temperature of application for optimum flexural strength. The incorporation of a surfactant into the wet fiberglass mat was found to result in improved resin penetration into the woven roving and increased flexural strength. The addition of 15 percent styrene to the presently used Port Hueneme resin (Selectron RS50338) and the incorporation of Triton X100 into the water in the fiberglass mat results in flexural strengths of 25,000 psi - a considerable improvement over the 4000 psi obtained with undiluted Port Hueneme resin. The influence of temperature was also investigated.

DERAKANE\* 510-A-40 when diluted with 15 percent styrene and, used with Triton X100, gives laminates having flexural strengths of 30,000 psi, meeting the 28,000 psi flexural strength specification. This resin does not have the desired five year shelf life.

This investigation was funded by the Civil Engineering Laboratory, Port Hueneme, California; under Contract No. N68305-77-C-0005.



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## GLOSSARY

Port Hueneme Resin

Resin received from Port Hueneme, Selectron RS50338 (PPG Industries, Inc.) Resin SR3704 plus 400 ppm dinitrophenol and 200 ppm p-toluhydroquinone.

Kirtland Primary Resin

Kirtland AFB resin 73% ALTEK B-25 and 24% DERAKANE\* 510, a brominated vinyl ester, 300 ppm added toluhydroquinone.

ALTEC (R) 8-52

Isophthalic polyester resin (Alpha Chemical Corporation); formulated from ALTEC 8-60 by adding styrene.

SELECTRON<sup>(R)</sup> SR3704

Polyester resin (PPG Industries, Inc.)

DERAKANE 510-A-40

Brominated vinyl ester formulated from DERAKANE 510-A by adding 40 percent styrene (The Dow Chemical Company).

Styrene

Styrene, 99%, inhibited 10-15 ppm p-tert-butylcatechol (Aldrich Chemical Company, Inc.)

**BPO** 

Benzoyl Peroxide

BZQ-40

A 40% benzoyl peroxide pourable paste (US Peroxygen Division, Witco Chemical Corporation).

CHP

Cumene hydroperoxide (Lucidol Division, Pennwalt Corporation)

CoN

Cobalt naphthenate, 6 percent cobalt (Nuodex Division of Tenneco Chemicals, Inc.)

DDM

LUPERSOL (R) DDM, a 60 percent solution of methyl ethyl ketone peroxide in dimethylphthalate (Lucidol Division, Pennwalt Corporation).

**DMA** 

N, N-Dimethylaniline.

**DMT** 

N, N-Dimethyl-p-toluidine (Aldrich Chemical Company, Inc.)

DNP

2,4-dinitrophenol (Pfaltz & Baurer).

<sup>\*</sup>Trademark of The Dow Chemical Company

MEKP

Methyl ethyl ketone peroxide

THQ

Toluhydroquinone (Eastman Chemical

Products, Inc.)

V

Vanadium TEN  $CEM^{(R)}$ , 4% Vanadium as vanadium neodecanoate (Mooney Chemicals, Inc.).

Promoter

Mixture of Vanadium TEN CEM and N,N-Dimethyl-p-toluidine

FABMAT (R) C-4020

Fiberglass mat composed of woven roving and chopped strand (Fiber Glass Industries, Inc.).

phr

Parts per hundred parts of resin.

#### SECTION I

## INTRODUCTION

The object of the work is to develop improved chemical components to be used in constructing fiberglass-reinforced plastic surfacings for soil in wet environments. The improved chemical components shall have shelf lives of five years minimum and shall be usable with the basic spray equipment now used by the Marine Corps. The work was funded by the Civil Engineering Laboratory, Port Hueneme, CA under Phase I of Contract No. N68305-77-C-0005.

Marine Corps requirements for rapid response and mobility call for methods by which Marine Corps engineers can upgrade the load-carrying capacity of native soils for temporary but often heavy vehicular and aircraft loads.

A recent development in soil surfacing techniques has been the fiberglass reinforced plastic (FRP) system called the Advanced Multipurpose Surfacing System (AMSS). The system pumps resin, catalyst and promoter through a hand-held spray gun, from which the materials are directed onto and into a fiberglass mat previously placed directly on the soil surface. Two resin hoses, one with added catalyst and one with added promoter, supply resin to two nozzles on the spray gun, combining the materials in two V-shaped fan sprays which intersect a short distance outside the nozzles. This system provides a promoted and activated resin. Further blending of these liquids occurs as a result of over-lapping passes of the spray on the fiberglass matting.

The fiberglass material presently in use is a woven roving with random fiberglass backing, supplied as FABMAT C-4020. This is a 40 oz. per square yard woven roving backed by 2 oz. per square foot of random fiber.

Application consumes about one pound of resin and associated components per square foot of fiberglass. Resin gelation time of about 10 minutes is obtained at working temperatures of over 40°F. One or two laminated layers of this FRP have been successfully utilized for expedient soil surfacings under a variety of vehicular loads over a wide range of soil conditions.

As constituted, however, the present system has the following deficiencies according to the Civil Engineering Laboratory:

- a. The FRP can be placed only under dry conditions at ambient temperatures above 40° F. Present Marine Corps requirements show a need for soil surfacings down to temperatures of -65° F and up to 140° F under wet conditions.
- b. The system now relies upon close tolerances in metering the flow of promoter and catalyst to provide a rapid (10 minutes) gelation time. Other, less sensitive techniques are needed to assure short gel times.
- c. The materials will propagate flames under some conditions. Fire retardancy and/or a self extinguishing capability are necessary features for use of the material in a combat environment.

Chemical components have been developed by the Government for formulating FRP soil surfacings that (1) have minimum shelf lives of 5 years, (2) are usable in constructing surfacings under dry conditions between ambient air temperatures of 40°F to 120°F, and (3) are usable with spray equipment now used by the Marine Corps.

In order to obtain the working life at 75°F of five years, a stabilized polyester resin, designated as RS50338, was developed. The new catalyst is cumene hydroperoxide (CHP); the promoter is a solution of vanadium neodecanoate (Vanadium Ten Cem) and N,N-dimethyl-p-toluidine. A retarder was developed, methyl ethyl ketone peroxide, that can be used to extend the gel time.

This work is specifically addressed to the development of a system of components to be used in the construction of fiberglass-reinforced plastic surfacings for soil under conditions where the fiberglass matting is completely wet with water (such as from a rainstorm) and the underlying soil is completely saturated with water.

## SECTION II

#### SUMMARY

The purpose of this investigation is to develop a chemical component system to be used for the field placement of a fiberglass-reinforced plastic soil surfacing under wet conditions, as from a rainstorm, where the ambient air temperature is between 32°F and 125°F. The components shall have a minimum shelf life of five years and shall be usable with the basic spray equipment now used by the Marine Corps. The contract requirement that the chemicals have a five year shelf life has, for all practical purposes, limited this work to resin systems previously developed for the Civil Engineering Laboratory under Contract No. N68305-76-C-0003, see Report No. CR77.017.

Reactivity studies were made of the resins used in this investigation. The control of the polymerization is more critical in the presence of water than when the fiberglass soil surfacing is prepared under dry conditions. The water acts as a heat sink and therefore more catalyst is required if a properly cured resin is to be achieved. The resin should not cure instantaneously as time is needed to allow the resin to displace the water from the fiberglass mat. The requirement of large amounts of catalyst to satisfy the heat capacity of the system and yet retard the gel time was accomplished by the use of methyl ethyl ketone peroxide, Lupersol (R) DDM. The resin used in most of this work was obtained from the Civil Engineering Laboratory and is a polyester resin produced by PPG Industries, Inc. as Selectron RS50338. This resin can only be properly cured by the use of a catalyst system composed of cumene hydroperoxide, vanadium neodecanoate (Vanadium Ten Cem), N,N-dimethyl-ptoluidine using methyl ethyl ketone peroxide as a retarder. The gel time can be varied from 10 to 30 minutes over the temperature range of 32°F and 125°F. The promoter composed of the Vanadium Ten Cem and the N, N-dimethyl-p-toluidine does have some limitations because variability of the Ten Cem from batch to batch and also because of age effects when it is formulated with the N,N-dimethyl-p-toluidine for use This requires that each batch of the as the promoter. Vanadium Ten Cem be calibrated.

Laminates were prepared using fiberglass mats and a hand lay-up technique that simulated the field preparation of the fiberglass plastic soil surfacings. The plastic reinforced laminates prepared under wet conditions were cured under 1 mm to 3 mm (0.039" - 0.118") of water. Flexural and tensile strengths were measured according to the appropriate ASTM test method.

The presence of only a small amount of water, 5% based on the dry mat) can result in a 70 percent loss in flexural strength. This is the amount of water that would result from the exposure of the FABMAT C-4020 for one hour to a 0.01 inch per hour rainstorm. A soil that is saturated with water has an equally deleterious effect on flexural strength. The effect of water in the substrate and in the fiberglass mat, on laminate properties, has been defined and solutions have been developed to some of the problems.

A low resin viscosity is necessary if the water is to be displaced from the fiberglass mats. The dilution of the Port Hueneme resin with 15 percent styrene results in much improved flexural strengths. When using this resin the incorporation of a nonionic surfactant, Triton X100, into the water in the fiberglass mat results in improved resin penetration and flexural strengths that are within 10 percent of specification. The brominated vinyl ester resin DERAKANE 510-A-40 when diluted with 15 percent styrene and used in conjunction with Triton X100 does give flexural strengths that meet the contract specification. It does not meet the contract shelf life specification.

The incorporation of the Triton X100 into the wet fiberglass FABMAT C-4020 will require an additional step in the constructing of fiberglassreinforced plastic surfacings of soils under wet conditions. A concentrated (10 percent) solution of Triton X100 in water would be sprayed onto the fiberglass mat. The mat would then be rolled to insure mixing of the surfactant with water in the mat before applying the resin.

#### SECTION III

#### TEST PROCEDURES

## A. ACCELERATED SHELF STABILITY TESTS

A three-dram vial filled to the shoulder with resin and capped with an aluminum-lined cap was used for testing. The resin volume and air space in the vial were held constant. Approximately 10 grams of resin were used, the exact weight depending on resin density. Care was exercised in testing the gel times because the gel first formed at the bottom of the vessel and a completely static test was avoided. However, oxygen in the air space can also reactivate the inhibitor to give falsely long stabilities, and therefore extensive agitation was avoided.

The vials were carefully inverted to minimize both the mixing of air into the sample and the formation of gel at the bottom of the vial. The samples were checked periodically from hourly at 120°C to daily at 80°C. The end point can be anticipated by the decrease rate of bubble rise as viscosity increases. The point at which the air bubble would no longer rise to the top of the vial was taken as the gel point or end of the test.

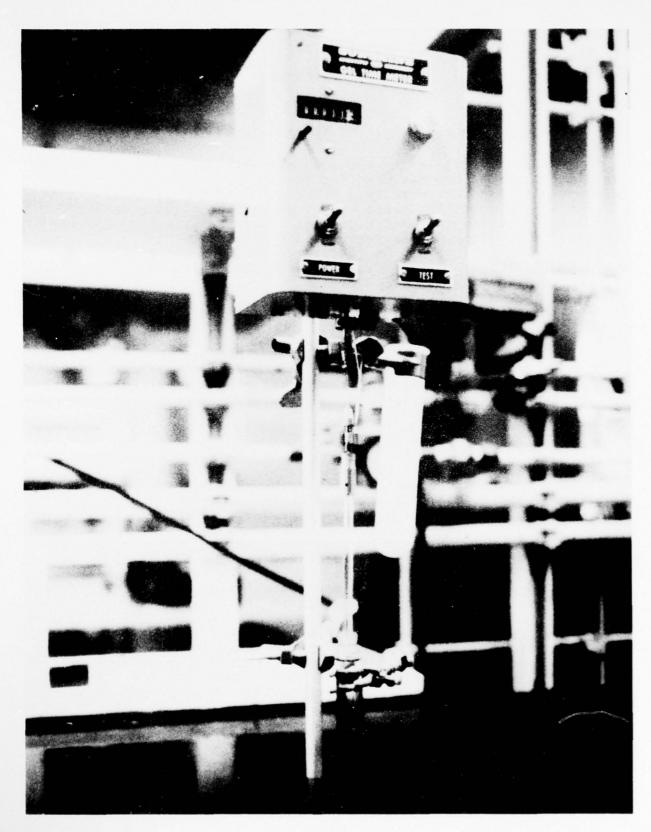
## B. REACTIVITY STUDIES

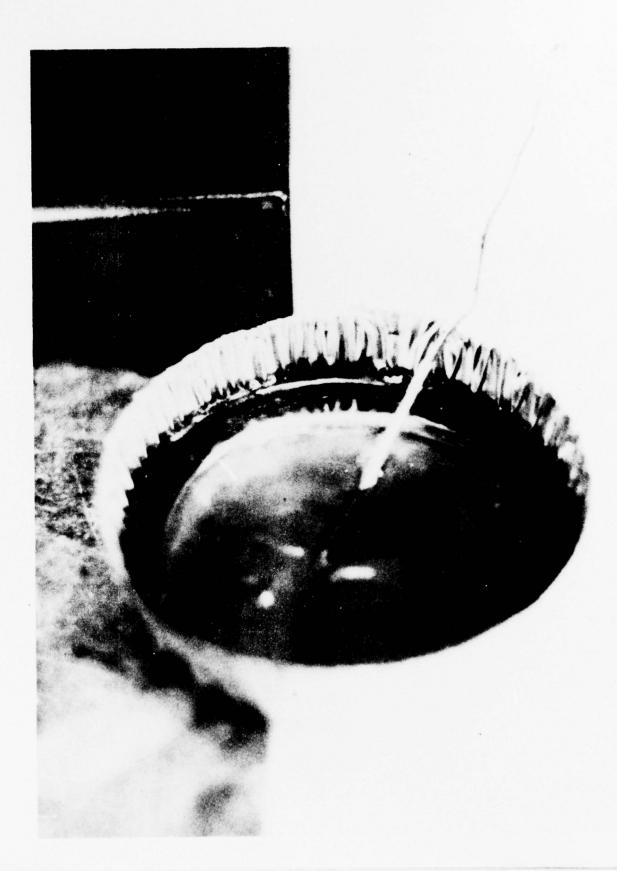
Reactivity testing consisted of determining the gel time, time to peak exotherm, temperature at peak exotherm, degree of cure by hardness (Barcol #934). Determinations were made with a Sunshine gel tester and with a time/temperature recorder.

Samples for the Sunshine gel tests were prepared by thoroughly mixing 10 grams of properly catalyzed resin and pouring the mixture into the Sunshine gel tube to the specified level. The switch controlling the spindle and timer was turned on and the gel time was recorded from the timer on the Sunshine tester (Figure 1).

Samples for the exothermic test were prepared by mixing 20 grams of properly catalyzed resin and pouring the resin in a 2-inch diameter aluminum foil dish. An iron-constantan thermocouple was imbedded in the resin (Figure 2) and attached to a temperature recorder. The changes in temperature were used to determine time to peak exotherm and temperature rise.

FIGURE 1
SUNSHINE GEL TESTER





## C. PREPARATION AND MECHANICAL TESTING OF GLASS LAMINATES

Laminates were prepared using three types of fiberglass mat chopped strand, woven roving, and FABMAT C-4020 at a resin to glass ratio of 60 to 40 by weight. Most of the work was done using two plies of 6 inch by 9 inch of FABMAT C-4020. The first ply of fiberglass was placed on the substrate, either a Mylar sheet or wet sand, into an adjustable frame made of 0.5 inch thick x 2 inches wide of the appropriate lengths of TEFLON to contain the mat. The frame was held in place by lead weights (see Figure 3). This frame was used to insure that when the laminates were prepared under wet conditions, the water did not run off. The predetermined amount of resin and catalyst at the appropriate temperature was then distributed over the fiberglass mat by pouring (Figure 3), and then rolled with a parallel groved roller until the resin was forced into the mat (Figure 4). In the case of the laminates prepared under wet conditions, the resin was allowed to exotherm before adding the next layer of FABMAT C-4020 and repeating the procedure. This was done to simulate field fabrication. The laminates prepared under wet conditions were cured under from 1 mm to 3 mm (0.039" - 0.118") of water, depending on the saturation of the mats. The laminate was cured at the appropriate temperature and then cut into test strips and tested on an Instron tester for flexural strength as specified in the ASTM procedure. In the case of tensile strengths the appropriate size specimens were milled from 0.75 inch strips using a Tensilcut milling machine and tested using the Instron following the specified ASTM method.





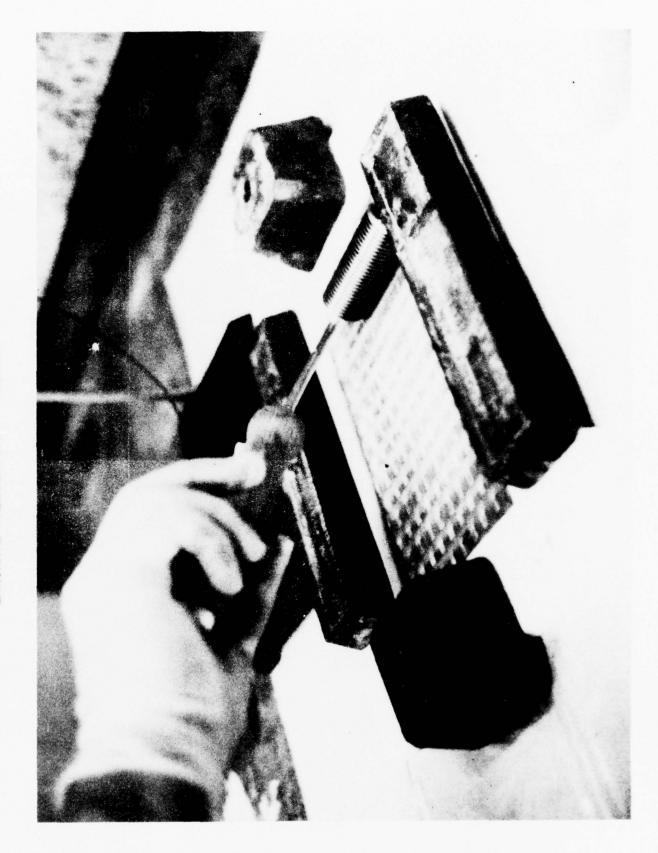


FIGURE 4

LAMINATE FABRICATION - ROLLING

#### SECTION IV

#### REACTIVITY STUDIES

The contract requirement that the chemical components "shall have shelf lives of (a) five years minimum for those components to be used in the wet environment" for all intents and purposes limited this work, to the use of the resin system developed under Contract No. N68305-76-C-0003, see report number CR77.017\frac{1}{2}. The catalysts used were cumene hydroperoxide (CHP) vanadium neodecanoate (V), methyl ethyl ketone peroxide (DDM), benzoyl peroxide (BPO), N,N-dimethyl-p-toluidine (DMT), cobalt naphthenate (CoN) and N,N-dimethylaniline (DMA). The polyester resin used by the Civil Engineering Laboratory at Port Hueneme and by the previous workers is manufactured by PPG Industries, Inc. All tests were made using resin supplied by the Civil Engineering Laboratory.

The gel time, a direct measure of reactivity, was obtained using 10 grams of catalyzed resin mixture in a Sunshine Gel Time Meter. At the same time the exothermic polymerization was followed by measuring the time and temperature behavior of 20 grams  $(0.25^{\circ})$  x  $(0.25^{\circ})$ 

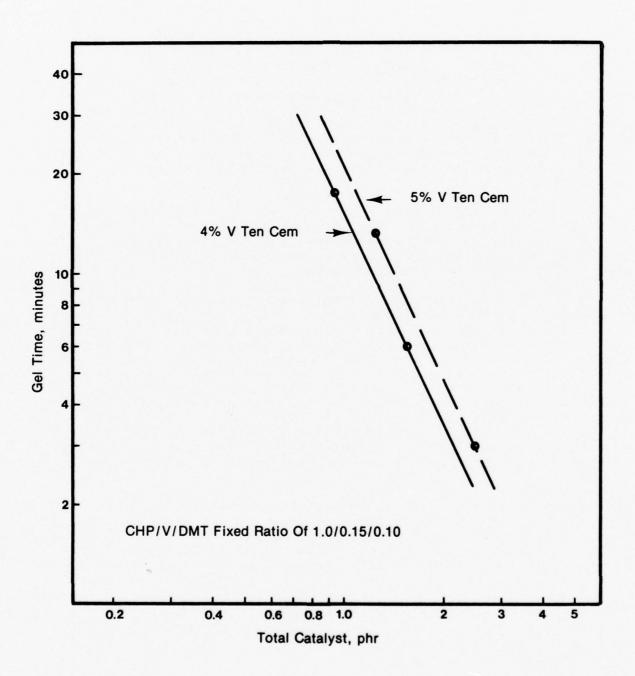
The 5% Vanadium Ten Cem used in the previous work is no longer manufactured by Mooney Chemicals, Inc. and is now only supplied as a 4% vanadium product. Although the product contains less vanadium, it is more active than the previous 5% product (see Figure 5). At a promoter (3p V:2p DMT) to cumene hydroperoxide ratio of 1 to 0.25 only 86 percent as much catalyst is required as with the previous 5% Ten Cem see Figure 5. In view of the extensive study made by Drakel showing that the optimum cure was obtained using a promoter composed of 3 parts of V Ten Cem to 2 parts of N,N-dimethyl-ptoluidine (DMT), most of this work was done using this ratio. The gel time was controlled by varying the total catalyst again using the previously determined cumene hydroperoxide to promoter ratio of 1 to 0.25.

The influence of water on the reactivity was determined by adding 20 grams of water to the 20 grams of resin in the aluminum dish and allowing the resin to cure under water. It was found that this results in an increased time to exotherm, due to increased heat capacity, and also decreased peak temperature and lowered barcol

<sup>&</sup>lt;sup>1</sup>Civil Engineering Laboratory, "Improved Chemical Components For Formulating Fiberglass-Reinforced Plastic Soil Surfacings," by S. S. Drake, H. E. Filter, D. L. Stevens, CR77.017, (May 1977, The Dow Chemical Comp. 19).

Figure 5

## **GEL TIME vs CATALYST CONCENTRATION**



hardness but a good cure was still obtained (see Table 1). The fact that the presence of water increases the time required for the resin to exotherm, because of the moderating effect of the increased heat capacity, made it obvious that the preparation of composites under wet conditions would require increased amounts of catalyst if the desired polymerization was to be attained. The use of increased amounts of catalyst results in rapid gelation and in the laboratory gives problems in composite fabrication that would not necessarily be encountered in the field. In the laboratory, the resin and catalyst are mixed in a cup and then poured onto Several minutes are required for this operation, while in the fiberglass. field applications instantaneous mixing is obtained. Therefore, to prevent large increases in resin viscosity, before incorporating the resin into the fiberglass the retarder, methyl ethyl ketone peroxide, Lupersol (R) DDM (DDM), was used. Reactivity studies were made using the polyester resin RS50338 as manufactured and with added styrene. The purpose of adding the styrene was to reduce the resin viscosity (see Section VD). It was found that the addition of styrene resulted in a change in reactivity (see Table 1 and Figure 6). Resin RS50338 containing an additional 10%, 15% or 20% styrene was found to have equal reactivity (see Figure 6). However, when the Lupersol DDM retarder is used, this is no longer the case (see Figure 7 and Table 1).

All of this investigation, except for the final stages, was made using the same batch of 4% V Ten Cem, Control S5335. The reactivity studies at 32°F (0°C) and 120°F (48.9°C) were made using another batch of 4% V Ten Cem Control LB-267-43. The composites made at 33°C and 120°F were also prepared using this sample. a variation in activity between these batches. Promoter made with the new batch of V Ten Cem has only slightly different activity than that obtained with the previous sample when used without Lupersol DDM. However, it it more sensitive to the inhibitor Lupersol DDM. In the case of Port Hueneme resin only 0.38 phr of DDM is required to obtain a gel time of 10 minutes using the new Ten Cem while 0.54 phr of DDM had been required previously. In both cases, the catalyst was composed of 2.5 phr cumene hydroperoxide, 0.375 phr 4% V Ten Cem and 0.25 phr N,N dimethyl-p-toluidine. The promoter made with this batch also showed a rapid change in activity with time (see Figure 8). When freshly made it was less active than after it had aged for two days. The gel time decreased from 11.3 minutes to 8.9 minutes after three days. No further change in activity was observed on an additional four days of aging. The reason for this behavior is not known but a similar effect of age had been observed by Drake 1. It is possible that the V Ten Cem and N, dimethylp-toluidine are not readily soluble in one another and that they slowly dissolve and increase in activity. To test this hypothesis the V Ten Cem and N,N dimethyl-p-toluidine were added separately and a gel time of 7.9 minutes was obtained as compared to 8.9 minutes obtained with the promoter that had aged seven days. This indicates that slow solubility or deflocculation might be the reason for the aging behavior. It is not known if Control Lot S-5335

Figure 6

## INFLUENCE OF STYRENE ON REACTIVITY

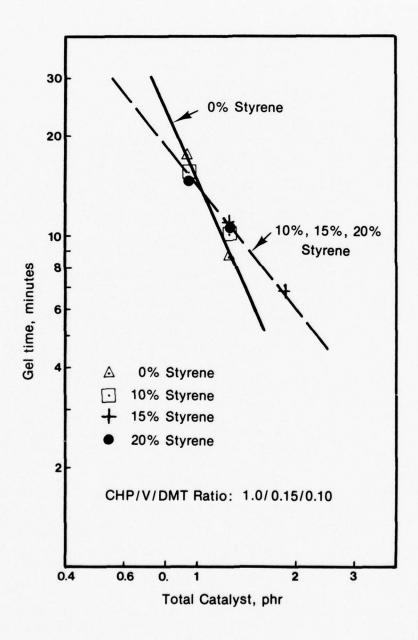


Figure 7

## INFLUENCE OF STYRENE ON REACTIVITY

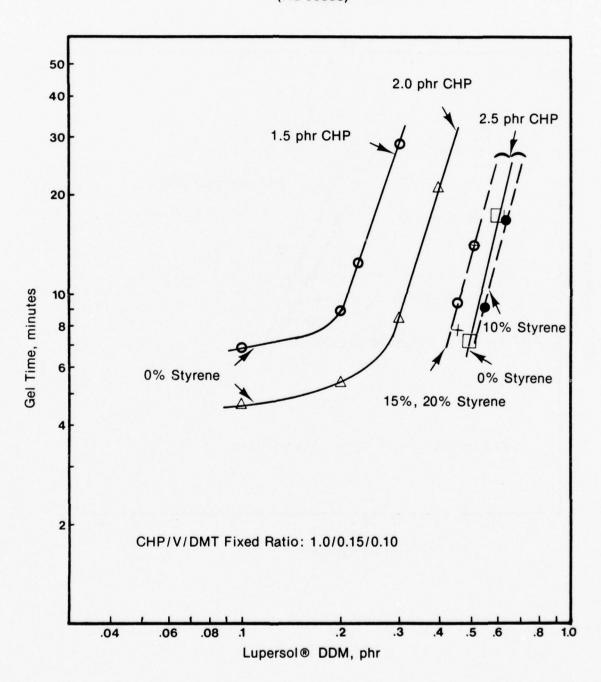
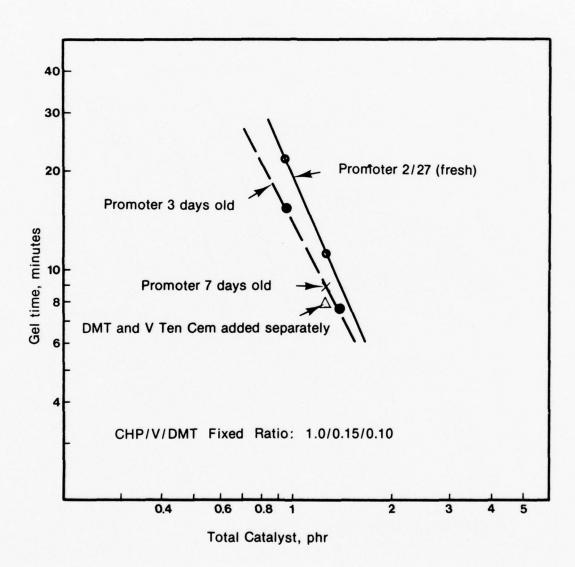


Figure 8

## EFFECT OF AGE ON PROMOTER ACTIVITY



showed a similar behavior but no change in activity was observed during its use. In any event the V Ten Cem LB-267-43 after two days aging has the same activity as S-5335. All activity tests that were made using a promoter prepared from LB-267-43 V Ten Cem were aged two days before use.

Mooney Chemicals, Inc., manufacturer of the Vanadium Ten Cem, was contacted, but could offer no explanation for the aging behavior. They stated that the vanadium neodecanoate is difficult to manufacture and that the activity does vary from batch to batch and is only manufactured to order.

The effect of changing the ratio of the promoter to cumene hydroperoxide as well as the promoter ratio on gel time is presented in Table 3, and Figures 9 and 10 for a promoter composed of 3 parts of 4% V Ten Cem to 2 parts of N, N\_dimethyl-p-toluidine and for a promoter composed of equal parts of the components.

The reactivity of Port Hueneme resin (RS50338) and Port Hueneme resin with 15% added styrene was determined at 32°F (0°C) and 120°F (48.9°C) (see Table 2 and Figures 11 and 12). As can be observed from Figure 12, the gel time obtained is a function of the heat transfer. AT 32°F, using the Port Hueneme resin with 15 percent added styrene, a gel time of 18.5 minutes was obtained when a liquid cooled jacket was used. The gel time decreased to 12.6 minutes when an air jacket was used. This is a result of less heat being removed with the air jacket giving decreased gel time. In the case of field application, under cold conditions it would be expected that the reactivity should approach the gel times obtained using a liquid jacket. At elevated temperatures, i.e., 120°F air temperature, the field application is more complicated in that the ground temperature through most of the day will be greater than the ambient air temperature resulting in shorter gel time. A plot for the selection of catalyst concentration over the temperature range from 32° F (0°C) to 120° F (48.9° C) is shown in Figure 13.

The reactivity of other resins than Port Hueneme polyester resin were also investigated. Tests were made of Selectron SR3703 (Port Hueneme RS50338 without added stabilizer) (see Table 4). The Kirtland Primary resin (76% Altek 8-52, an isophthalic polyester resin, and 24% DERAKANE 510, a brominated polyester) were also investigated (see Table 5 and Figure 14). In order to study the effect of resin density as well as viscosity on the preparation of wet composites the reactivity of DERAKANE 510-A-40 was investigated using various catalysts (see Tables 6 and 7 and Figure 15). The DERAKANE 510-A-40 is not as active as the Port Hueneme resin and becomes less active on the addition of 15% styrene (see Tables 3, 8 and Figure 15).

Figure 9

## INFLUENCE OF PROMOTER RATIO ON REACTIVITY

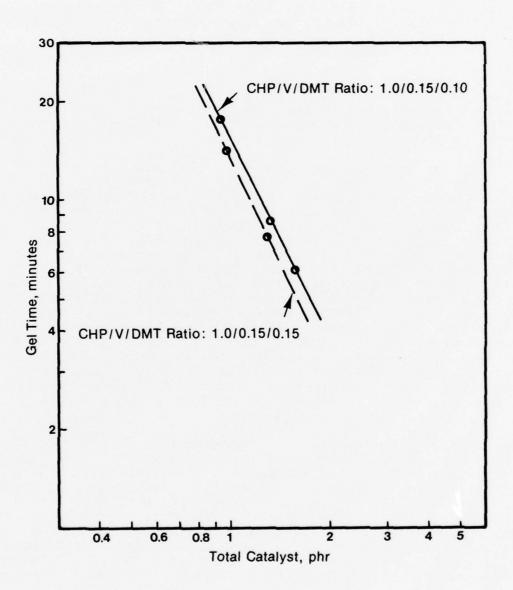


Figure 10

## EFFECT OF PROMOTER ON GEL TIME

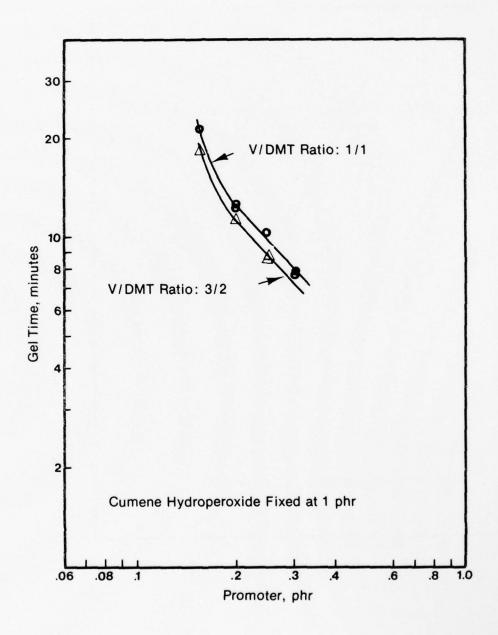


Figure 11

## GEL TIME vs CATALYST CONCENTRATION AT DIFFERENT TEMPERATURES

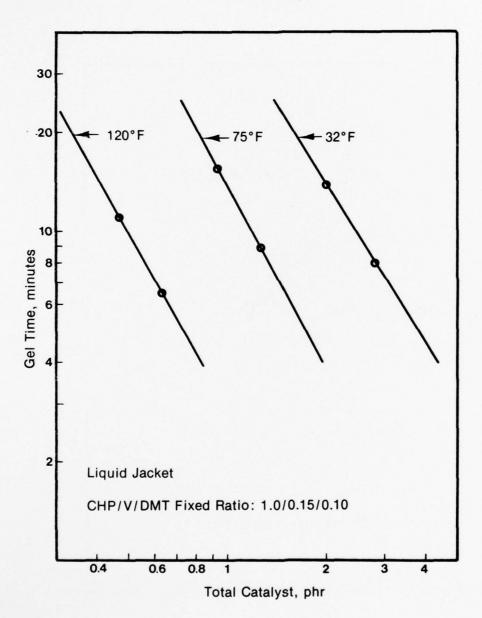


Figure 12

# GEL TIME vs CATALYST CONCENTRATION AT DIFFERENT TEMPERATURES

## PORT HUENEME RESIN + 15% STYRENE

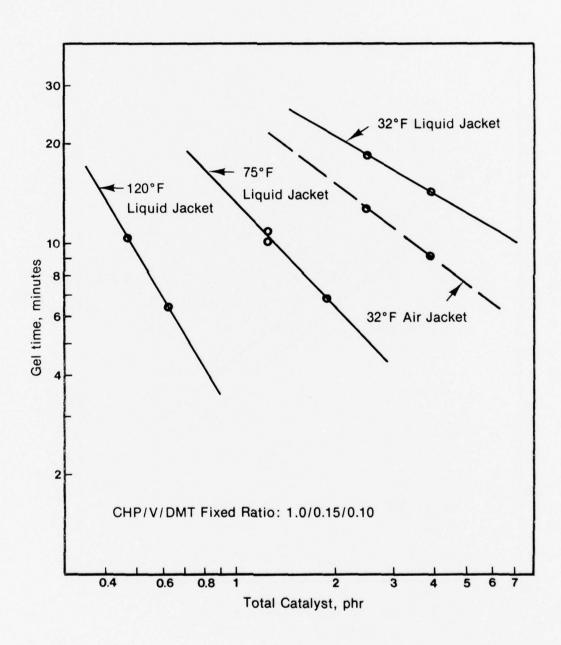


Figure 13

# SELECTION OF CATALYST CONCENTRATION vs TEMPERATURE OF APPLICATION

PORT HUENEME RESIN (RS 50338)

(Constant Gel Time, 10 minutes)

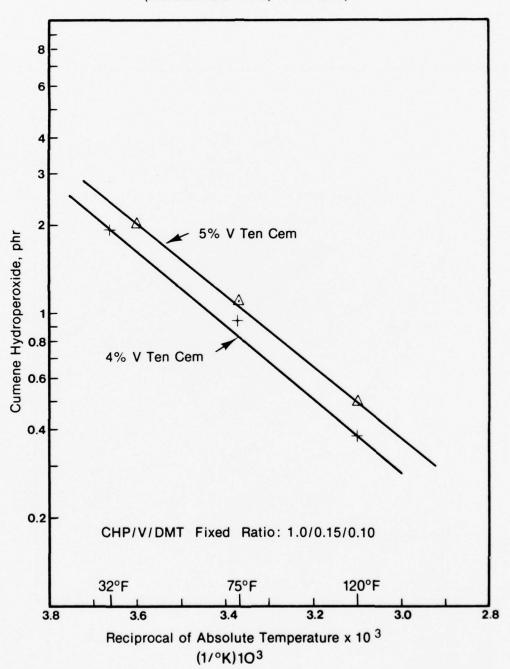


Figure 14

## EFFECT OF RETARDER

## KIRTLAND PRIMARY RESIN

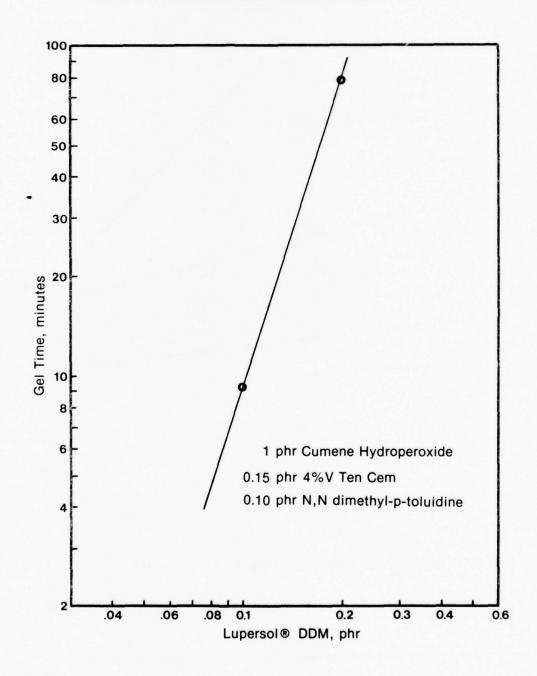
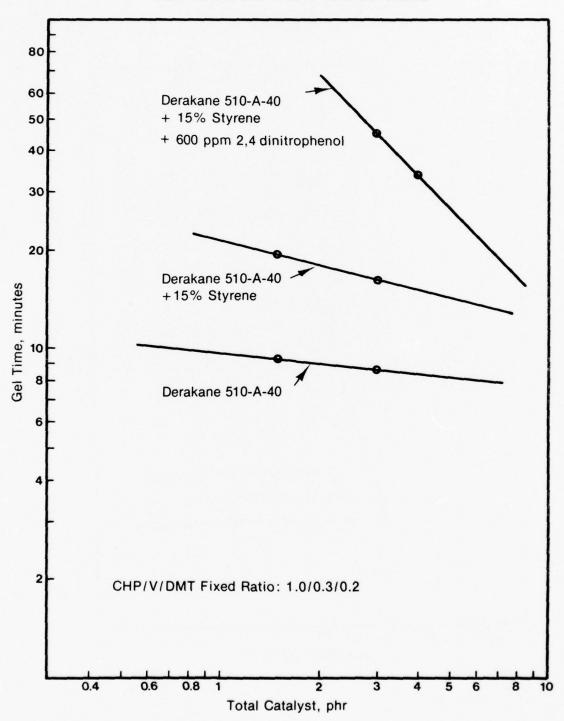


Figure 15

# REACTIVITY DERAKANE 510-A-40 DERAKANE 510-A-40 + 15% STYRENE



#### SECTION V

## PREPARATION OF FIBERGLASS REINFORCED PLASTIC LAMINATES UNDER WET CONDITIONS

As discussed previously, the requirement that the chemical components have a five year minimum shelf life restricted most of the work to an effort to modify the previously developed resin system to wet conditions. Most of the fiberglass laminates were made using the fiberglass material presently used by the Civil Engineering Laboratory at Port Hueneme. This is a woven roving with a random fiberglass backing manufactured by Fiberglass Industries, Amsterdam, New York. This is a 40 oz. per square yard woven roving backed by 2 oz. per square foot of random fiber and is designated as FABMAT C-4020 by the manufacturer.

## A. EFFECT OF WATER ON FLEXURAL STRENGTH

The study of the influence of water on the flexural strength of fiber-glass laminates was made using two plies of 6 inch by 9 inch FABMAT C-4020. These fiberglass laminates were prepared using the hand lay-up technique used in the previous work done for the Civil Engineering Laboratory and is described in detail in the test section. The fiberglass mats were wet using two different methods. The low moisture mats (10% moisture based on the dry mat weight) were prepared by carefully misting the alternate sides of the mat until the desired water content was attained. The higher water content fiberglass mats were prepared by covering with distilled water and then draining, if necessary, on paper towels. The catalyst, promoter and inhibitor were varied with the moisture content to insure adequate polymerization. The results obtained are presented in Table A (see Table 9).

TABLE A

EFFECT OF MOISTURE ON FLEXURAL STRENGTH

0/0	Moisture*				F	lexural Strengt Lbs/sq in.	h
	0					39,600	
	4.8 10.2					10,800	
	25.8 25.8					13,000 11,700	
	27.4 50.4					13,460 6,100	
	71.4					3,600	
* (	71.7 % Moisture =	wt.	wet	mat	- wt.	3,900 dry mat x 100	

\* % Moisture =  $\frac{\text{wt. wet mat - wt. dry mat}}{\text{wt. dry mat}} \times 100$ 

As might be expected, even a small amount of moisture, 4.8 percent has a most harmful effect on the flexural strength of the laminate resulting in a 70 percent loss in strength. This is the moisture content that would result from a one hour exposure of the FABMAT C-4020 to a 0.01 inch per hour rainfall. Increasing the moisture content to 27.4 percent has no significant effect on the flexural strength but higher moisture levels do result in a further loss in flexural strength (see Table A). The results indicate that the major reason for the loss of flexural strength of laminates made under wet conditions is due, for the most part, to the inability of the resin to bond to the wet fiberglass mat, not to poor resin The maximum amount of water that the FABMAT C-4020 will hold is 70 percent by weight of mat and this amounts to about 0.45 cc of water per cc of resin. To put it another way, at a 70 percent water content the fiberglass mat would be holding the maximum amount of water that it could contain. In all cases, placement of the resin resulted in free water covering the resin surface so that, in effect, all of the tests made at 70 percent water in the fiberglass mat were made under 3 mm (0.11 inches) of water.

#### B. EFFECT OF SUBSTRATE ON FLEXURAL STRENGTH

The influence of water in the soil under the fiberglass was investigated. It was found that when fiberglass laminates were made using 100 percent water-saturated sand as a substrate this had a most deleterious effect on the flexural strength. The flexural strength decreased from 36,000 psi obtained with dry fiberglass laminates and a Mylar substrate to 4,600 psi - 8,600 psi with the saturated sand substrate.

A laminate was also prepared on the water saturated sand using fiberglass mats containing 70.4 percent water. A flexural strength of 4,450 psi was obtained (see Table 10). This compares to the 3,700 psi obtained with mats containing 71 percent water and a Mylar substrate. This shows that when a laminate is prepared from wet fiberglass mats on saturated sand that further loss in flexural strength does not result. Using dry sand as a substrate flexural strengths of 36,700 psi were obtained. The loss in strength that results when the laminate is prepared on the wet substrate is as great as when the fiberglass is wetted. Therefore, the loss in strength must be due to the wicking of the water into the fiberglass mat. Until this time the fiberglass laminates had been prepared by placing the fiberglass mat on the substrate, pouring the appropriate amount of resin onto the fiberglass mat and then rolling; followed by the immediate placement of the second mat, etc. This technique is satisfactory under dry conditions. It does not, however, simulate field practice. Under wet conditions, the fabrication technique might have an influence on the fiberglass laminate properties as a result of different dispersion of the water in the resin, water migration may be different dependent on the mat placement, i.e., woven roving up or down, wicking from the soil, etc. In order to more nearly simulate the field application of fiberglass laminates.

the following laboratory fabrication method was used, hereafter referred to as the sequential method. In this technique after applying the resin to the first fiberglass mat the resin was allowed to exotherm before applying the second fiberglass mat and resin since this is the way it is done in the field. This change in the method of fabrication (allowing the resin to exotherm between layers) resulted in the flexural strength increasing to approximately 15,000 psi. No doubt due to the fact that moisture only entered into the first fiberglass mat. It was thought that placing the cut strand side of the FABMAT C-4020 in contact with the wet sand rather than the woven roving side would result in less wicking and improved flexural strength but no improvement resulted (see Table 10) In a further effort to decrease the wicking of water into the mat the FABMAT C-4020 was treated with dichlorodimethylsilane to increase the hydrophobicity. This treatment did not result in any improvement in the flexural strength of the laminate prepared on the wet substrate.

## C. INFLUENCE OF FABRICATION METHODS ON FLEXURAL STRENGTH

Since the method of fabrication of the fiberglass laminates on 100 percent water saturated sand did have an effect on the flexural strength a study was made to more completely insure that the proper laboratory technique was used. In the field preparation of fiberglass laminates the FABMAT C-4020 is placed on the soil with the woven roving side down and the resin and catalyst are sprayed onto the mat and rolled. After the resin has exothermed the second layer of mat is spread, again with the woven roving side down, and the resin applied and rolled. The same method of preparation was followed in the laboratory. Tests were made to determine if this sequential method of allowing the resin to exotherm between layers and if the direction of placement of the woven roving would have any influence on the flexural strength of laminates. As would be expected when dry fiberglass mats were used the method of fabrication, sequential or non-sequential, nor the placement of the woven roving, either up or down, had any effect on the flexural strengths (see Table 11). All of the flexural strengths were determined with the specimens placed in the test machine so that the woven roving was on the side of maximum stress. Tests were also made where two layers of wet fiberglass mat were used. Again, the placement of the woven roving side, either up or down, was not found to have any significant influence on the flexural strength using mats containing 25 percent water. Nor did allowing the resin to exotherm before applying the second mat have any influence on strength.

In the work reported in a previous section, it had been observed that the laminates prepared using Port Hueneme resin and FABMAT C-4020 under wet conditions had shown what appeared to be very poor

penetration of the resin into the fibers of the woven roving. The inability of the resin to displace the water trapped in the strands of the continuous fiber is no doubt the reason for the marked loss in flexural strength in the presence of relatively small amounts of water (5-10%). In an effort to confirm that the lack of resin penetration into the woven roving is the major reason for the loss in strength, laminates were prepared using only chopped strand mats. The use of the chopped strand mats should insure good resin penetration. Piberglass laminates were prepared by placing 3 layers of 2 oz/ft chopped strand (either wet or dry) on the Mylar sheet. Then the desired amount of resin and catalyst was added and the layer rolled. After the resin had exothermed, a second layer was placed on top of the cured resin. The reason for using three of the chopped strand mats in each layer was to have about the same amount of glass in the finished laminate as in the case of the FABMAT C-4020 laminates. The following results were obtained:

% Water	Flexural Strength
0	21,750 psi
25.5	18,360 psi
26.0	19,530 psi
69.8	14,440 psi

These results are markedly different from those obtained with FABMAT C-4020. The presence of approximately 26 percent water in the chopped strand laminates resulted in only a 13 percent loss in flexural strength as compared to up to a 60 percent loss in flexural strength with the laminates prepared with the FABMAT containing the woven roving. Increasing the water content of the chopped strand mats to 69.8 percent resulted in only a 34 percent loss in flexural strength as compared to the 90 percent loss in flexural strength obtained when the FABMAT C-4020 was used. As further confirmation laminates were prepared using two layers where each layer was composed of two layers of 18.5 oz/yd of woven roving and one layer of 2 oz/ft of chopped strand. In the case of the laminates the no water flexural strength was 41,000 psi (see Table 11) and when 24.5 percent water was in the fiberglass a strength of 9,830 psi was obtained. This is a 76 percent decrease in flexural strength.

A test was also made using a single layer of FABMAT C-4020. The no water average flexural strength was 22,910 psi and when 70.6 percent water was in the mat,the strength was 1,860 psi,a decrease of 92 percent. These results seem to confirm the conclusion that the major effect of water is to prevent the penetration of resin into the woven roving and not resin cure or resin adhesion. If it were poor cure at the fiber surface resulting in poor adhesion, the chopped strand laminates should have shown greater decreases in flexural strength than the 13 percent obtained at 26 percent water content and the 34 percent at 69.8 percent water.

In view of the relatively small loss in strength obtained with laminates from the wet chopped strand fiberglass mats an effort was made to obtain a spun strand woven roving. In the presently used FABMAT C-4020 the woven mat is manufactured from continuous strand roving - parallel glass strands. The spun strand roving is also made from continuous filament strands in which the continuous strands are looped back and forth upon themselves and held in roving form by a slight twist and by the use of a resinous size. This is a more bulky roving and would no doubt result in lower dry strength laminates. The converse should be true for laminates prepared under wet conditions. The more open nature of the spun woven roving should result in better resin penetration and increased strength. Efforts were made to obtain such a fiberglass mat but at this time no supplier has been located.

## D. EFFECT OF RESIN VISCOSITY AND SURFACTANTS ON FLEXURAL STRENGTH

The previous work has shown that if satisfactory flexural strengths are to be obtained using FABMAT C-4020 mats that better resin penetration into the woven roving must be obtained. One way to accomplish this is to decrease the viscosity of the Port Hueneme resin by dilution with styrene. In doing this not only is the viscosity reduced but at the same time the resin density is decreased. The lowered resin density will result in poor water displacement as the resin density approaches the water density.

In the first test that was made, the Port Hueneme resin was diluted with an additional 10 percent of styrene. This resulted in the viscosity decreasing from 218 cps to 109 cps and at the same time the density decreased from 1.093 g/cc to 1.07 g/cc. As would be expected, the addition of the styrene resulted in changed reactivity (see reactivity section of this report). Laminates were made using the FABMAT C-4020 fiberglass mats containing 25 percent water. Flexural strengths ranging from 20,850 psi to 23,450 psi (average 22,150 psi) were obtained (see Table 12). This is a significant improvement over the 13,000-16,000 psi flexural strengths obtained with laminates prepared from mats containing 25% water and with Port Hueneme resin without additional styrene. A laminate was also fabricated from fiberglass mats containing 70.8 percent water. A flexural strength of 12,440 psi, with the Port Hueneme resin with added 10 percent styrene, was obtained as compared to 4000 psi with Port Hueneme resin. It is felt these improvements in flexural strength are due, for the most part, to improved resin penetration into the woven roving as a result of the lower resin viscosity. These laminates were prepared using two different types of rollers to determine if the resin could be mechanically forced into the woven roving to displace the water. A paddle roller was used in the hope that its kneading action would result in better water displacement than the normally used vertical disk roller. No significant difference in flexural strength resulted from the use of these different type rollers in the preparation of the laminates.

In view of these results where the addition of 10 percent styrene (by weight) to the Port Hueneme resin (RS50338) resulted in increased flexural strength under wet conditions; the effect of further dilution with styrene was investigated (see Table 12, and the following Table B).

TABLE B

Effect of Styrene Added to Port Hueneme Resin on Flexural Strength

Styrene*	Viscosity cps	Density g/cc	Water %	Flexural Strength-psi
0	218	1.093	70	4,000
10	109	1.07	70.8	12,440
15	80	1.06	70.9	18,770
20	5.5	1.05	70.3	12,340
*% styre	ene = wt. sty	rene x	100	
	wt. of	RS50338 resi	n	

As can be seen, the optimum flexural strength is obtained on the addition of 15 percent styrene to the Port Hueneme resin. This results in a flexural strength of 18,770 psi for a laminate prepared from mats containing 70 percent water; further dilution results in decreased strength. The dilution of the resin with styrene had no effect on the flexural strength of laminates prepared from dry mats being  $42,000 \pm 2,000$  psi. Although the addition of styrene has resulted in a 470 percent increase in flexural strength to 18,700 psi, it is still 10,000 psi short of the desired flexural strength of 28,000 psi.

Another approach to obtaining increased flexural strengths is to treat the water so that it is more easily displaced by the resin. This could be done by adding an appropriate surfactant. This would result in not only a decrease in the interfacial tension, but also in the surface tension of the water. The first tests were made using Triton X100, a water-soluble isooctyl phenoxy polyethoxy ethanol. This surfactant was chosen because it is non-ionic and should have no influence on the reactivity and did not. It also has the property of being biodegradable. The first tests were made by wetting the mat to 25 percent water and then spraying the mat with a one percent solution of Triton X100 until the mat contained 5 to 8 percent (by weight) of the 1 percent solution (final surfactant concentration being 0.17 - 0.24 Triton X100). Flexural strengths of 17,000 psi were obtained, only a slight improvement over water alone but enough to indicate that this approach had promise. A laminate was then made on which the FABMAT was wet with the one percent Triton X100

in water and gave a flexural strength of 25,280 psi (see Table 12) at 25 percent fluid as compared to 13,000 psi with water. On increasing the moisture content to 69.6 percent using the surfactant solution a flexural strength of 17,670 psi was obtained as compared to 4000 psi with water alone. This significant increase in strength shows that the addition of a surfactant, to change the surface tension and interfacial tension, does result in better displacement of the water from the fiberglass mat.

Laminates were prepared using Port Hueneme resin containing 10 percent styrene. Flexural strengths of 23,080 psi were obtained at 24.1 percent saturation and 24,610 psi at 68.1 percent saturation with Triton X100 solution. At 15 percent styrene and 70 percent surfactant saturation the flexural strength averaged 25,080 psi. These flexural strengths approach the desired 28,000 psi. Indications are that 1% Triton X100 is more than is required since reducing the concentration to 0.5% did not result in a significant change in flexural strength, 17,670 psi to 16,820 psi, in the case of Port Hueneme resin with 70% mat saturations.

If surfactants are to be used in the field the method of application would be to spray a concentrated solution of the surfactant, in water, onto the wet mat and then allow the surfactant to mix with the water in the mat either by diffusion alone or by mixing with a roller or some other mechanical method. The effectiveness of diffusion was determined using Port Hueneme resin diluted with 15 percent styrene (Table 12). The mat containing 69.7 percent water was sprayed with a 10 percent Triton X100 solution (12.5 percent added based on dry mat) onto the wet mats and allowed to stand thirty minutes before adding the resin. In this case a flexural strength of 19,660 psi was obtained as compared to 23,320 psi when the mats contained 70 percent of one percent Triton X100. Although the total fluid in the mats was greater in the diffusion test, 82.2 percent as compared to 70.4 percent, the sixteen percent loss in flexural strength indicates that some mechanical mixing would be desirable. This was done in a similar way except that after spraying the 10 percent water solution of Triton X100 onto the wet mat, the mat was then rolled with the same roller that is used to distribute the resin. Again, the fiberglass mats contained about 70 percent water and from 5.1 to 11.1 percent (by weight of dry mat) of the Triton X100 solution was sprayed onto the mats. The flexural strengths obtained averaged 15,610  $\pm$  1,050 psi in this case as compared to 17,200  $\pm$ 400 obtained with a 1 percent Triton X100 solution in the mats (see Table 12). These tests were made using Port Hueneme resin. The results show that the beneficial effect of a surfactant can be obtained by spraying a concentrated solution onto the water saturated FABMAT C-4020 followed by rolling.

It was not possible because of the contract time limitations to undertake a surfactant optimization investigation. Some tests were made using other surfactants. Triton CF-76, a nonionic detergent, appeared to be more effective than the Triton X100 in the case of

Port Hueneme resin giving a flexural strength of 22,860 psi as compared to the 17,200 psi obtained with Triton X100. Both tests were made using mats containing 70 percent of a one percent surfactant solution (Table 12). However, the Triton CF-76 does not offer any advantage over Triton X100 when used in conjunction with the less viscous Port Hueneme resin containing 15 percent styrene; 25,800 psi flexural strength compared to 25,080 psi with Triton X100. Pyronate 50, a water-soluble petroleum sulfonate, was tested using fiberglass mats containing 72.4 percent of a one percent solution. It was found that under these conditions the Port Hueneme resin did not cure. In view of this the use of ionic surfactants does not appear to be promising. At low water saturations (24%) spraying the mat with ethyl alcohol (2B formulation) was found to have a beneficial effect on flexural strength, but at 70 percent water the converse is true. A laminate prepared using mats containing 70.3 percent water and then spraying with alcohol (13.2% by weight of dry mat) had a flexural strength of 10,360 psi using Port Hueneme resin containing 15 percent styrene. Under these conditions alcohol is not effective.

# E. EFFECT OF TEMPERATURE ON LAMINATE PROPERTIES

Studies were made at 33°F (0.6°C) and 120°F (48.9°C) of the effect of temperature on the flexural strength of laminates prepared using Port Hueneme resin and Port Hueneme resin containing 15 percent styrene (see Table 14). At the same time the influence of temperature on the viscosity of these resins was also obtained (see Table 16). All materials used in preparation of the laminates were pre-heated or cooled to temperature and the laminates were held at this temperature overnight. Samples were cut and the flexural strengths determined within two hours after cutting so that the low temperature laminates would not give false results (see Table C). expected, the laminates prepared with wet mats at 33°F had low flexural strengths; 4,290 psi with Port Hueneme resin and 9,240 psi with Port Hueneme resin containing 15% styrene. This is a result of two factors: one of increased resin viscosity (see Table 2) that results in poor penetration into the woven roving and second, poor resin cure. Conversely, increasing the temperature to 120° F resulted in good resin penetration, cure and flexural strengths of 33,410 psi to 35,470 psi for these resins with mats containing 70 percent water (see Table C).

TABLE C

Effect of Temperature on Flexural

Strength of Laminates

Resin	%* Water	%* Surfactant		ength psi	Rem	arks	
Temperature 33°F	(0.6°C).						
Port Hueneme Port Hueneme +15% Styrene	0	74.1 72.3				Triton Triton	
Temperature 120°	F (48.9°C)						
Port Hueneme Port Hueneme Port Hueneme +15% Styrene	73.4 0 0	0 70.2 71.0	Flex.		1%	Triton Triton	

<sup>\*%</sup> Water or surfactant =  $\frac{\text{wt of water or surfactant}}{\text{dry wt of mat}}$  x 100

These results indicate that at a high ambient temperature (120°F), Port Hueneme resin will give satisfactory strengths when used under wet conditions and the use of surfactants will not result in any increase in flexural strength.

Tensile strengths were determined for Port Hueneme resin, Port Hueneme resin containing 15 percent styrene and DERAKANE 510-A-40 containing 15 percent styrene under wet conditions, 70 percent water. with and without Triton X100 and at 75°F (23.9°C). The lowest tensile strength obtained was 16,630 psi to a high of 26,300 (see Table 15). In all cases, the tensile strength was greater than the 13,000 psi specified in the contract.

#### F. INFLUENCE OF RESIN DENSITY

As discussed previously, if satisfactory flexural strengths are to be obtained it is necessary that the resin penetrate the woven roving and displace the water. In order to accomplish this, the resin must be fluid with low viscosity, and also it should be dense since gravity acting on the density difference between the resin and the water

supplies the driving force. This is supplemented by mechanical work supplied by the roller on the fluid filled fiberglass mat. One of the most dense resins available is a brominated vinyl ester resin DERAKANE 510-A-40 manufactured by The Dow Chemical Company. This resin has a density of 1.22 as compared to 1.093 for Port Hueneme resin. The first tests were made using the recommended methyl ethyl ketone peroxide catalyst but the laminates would not cure properly under wet conditions. The catalyst was changed to cumene hydroperoxide and the Port Hueneme resin promoter with which satisfactory cures were obtained. Laminates prepared at 25 and 70 percent water had flexural strengths of 15,240 psi and 14,310 psi, respectively. This resin did not show the desired result of high flexural strength due to density. Surprisingly, little change in strength resulted with increased water saturation. The DERAKANE 510-A-40 was then diluted with 15 percent styrene to reduce the viscosity from 335 cps to 85 cps and the density from 1.22 to 1.17. Laminates made with DERAKANE 510-A-40 + 15% styrene had a flexural strength of 30,000 psi at 25 percent water (Table 12). and 24,160 psi at 71.4 percent water. Laminates prepared from mats containing 28.6 and 68.8 percent of a one percent solution of Triton X100 gave flexural strengths of 26,570 psi and 30,300 psi, respectively. The influence of further dilution of the DERAKANE 510-A-40 with styrene was continued using DERAKANE with an additional 20 percent styrene. Flexural strengths of 25,390 psi were obtained with laminates prepared from fiberglass mats containing 70 percent water and 29,130 psi from mats containing 70.2 percent of one percent Triton X100; no improvement over the DERAKANE 510-A-40 plus 15 percent styrene. The results obtained with the DERAKANE 510-A-40 indicate that low resin viscosity is more important in obtaining good resin penetration into the woven roving than resin density. The DERAKANE 510-A-40 plus 15 percent of styrene gives flexural strengths that are at the low end of the flexural strength specification without the use of a surfactant. When used in conjunction with Triton X100 it produces laminates that meet specifications for flexural strength. It does not meet the five year shelf life specification and when diluted with styrene shows poor reactivity see previous section on reactivity. It is the best resin tested for use under wet conditions and it and the other vinyl ester resins should be investigated further for use under wet conditions.

#### G. MISCELLANEOUS RESINS

Selectron SR3703 (Port Hueneme resin without added stabilizer) was investigated to determine if a more reactive resin would give improved strengths under wet conditions. The SR3703 when used with fiberglass mats containing 5 percent water gave laminates with flexural strengths of 20,000 to 26,000 psi an increase of about 50 percent over the Port Hueneme resin (Table 13). When the water content in the mats was increased to 25 percent the flexural strength obtained with the SR3703 laminates were equal to, but no better than,

that obtained with Port Hueneme resin. The Kirtland Primary resin (76% Altek 8-52 and 24% DERAKANE 510) was also evaluated and showed no advantage over Port Hueneme resin when used under wet conditions (see Table 13).

#### H. FIBERGLASS MAT TREATMENT

In an effort to obtain improved flexural strengths under wet conditions the FABMAT C-4020 was treated with various organic silane compounds to increase the hydrophobicity of the fiberglass. If the glass fibers could be made more hydrophobic, this should result in better resin penetration into the woven roving and improved flexural strengths. The FABMAT C-4020 was treated with dichlorodimethylsilane, octadecyltrichlorosilane and diphenyldichlorosilane. The fiberglass mats were silaned by both vapor and liquid phase treatment. The results obtained are presented in Table D.

TABLE D

Effect of Silane Treatment of FABMAT C-4020 on Flexural Strength

Run No. Port Huen	% Water eme Resin +	Flexural Strength, psi 10% Styrene	Remarks
0105B	25.3	24,420)	Mat treated, vapor phase
0206A	70.8	16,880)	octadecyltrichlorosilane
0111C	0	17,330)	Mat treated with octa-
0112A	24.9	21,080)	decyltrichlorosilane in toluene
Port Huen	eme Resin +	15% Styrene	
0109B	24.5	17,400)	Vapor phase octadecyltri-
0109C	0	18,060)	chlorosilane
0126A 0206A	25.8 70.8	27,500) 16,880)	Vapor phase diphenyldichlorosilane

In no case did an increase in flexural strength result from the use of silane treated fiberglass as compared to laminates prepared from untreated FABMAT C-4020.

#### SECTION VI

## ACCELERATED SHELF LIFE STUDIES

Accelerated shelf life studies of Port Hueneme resin and Port Hueneme resin with 15 percent added styrene have been made. This was done to determine if the addition of styrene to the Port Hueneme resin would have an adverse effect on the shelf life at 75° F. According to the work of Filter and Lard, the shelf life at 75° F for any given resin system can be predicted from the elevated temperature studies. A straight line is obtained when the shelf life is plotted against the reciprocal of the absolute temperature and the point at which this line intercepts the 75° F line is the 75° F shelf life. Shelf lives predicted from accelerated aging studies based on field experience are conservative. The following shelf lifes were obtained:

Resin	She1f	Life Hours
	80°C	120°C
Port Hueneme	269	10
Port Hueneme + 15% styrene	262	9

The shelf life vs reciprocal of the absolute temperature plot is given in Figure 16. The addition of the 15 percent styrene has a negligible effect on the shelf life of this polyester resin since both resins have estimated shelf lives of over 11 years. The fact that the Port Hueneme resin had an estimated shelf life this long was rather surprising since at the time of the initiation of the test the resin had been in outside storage at Port Hueneme for over a year.

The DERAKANE 510-A-40 vinyl ester resin and DERAKANE 510-A-40 containing 15 percent styrene were also investigated and were found to have shelf lives of less than one hour at 120°C. Toluhydroquinone (THQ) and 2,4 dinitrophenol (DNP) were then tested as stabilizers for the DERAKANE 510-A-40 containing 15 percent styrene at 120°C. The following shelf lives were obtained (see Figure 16):

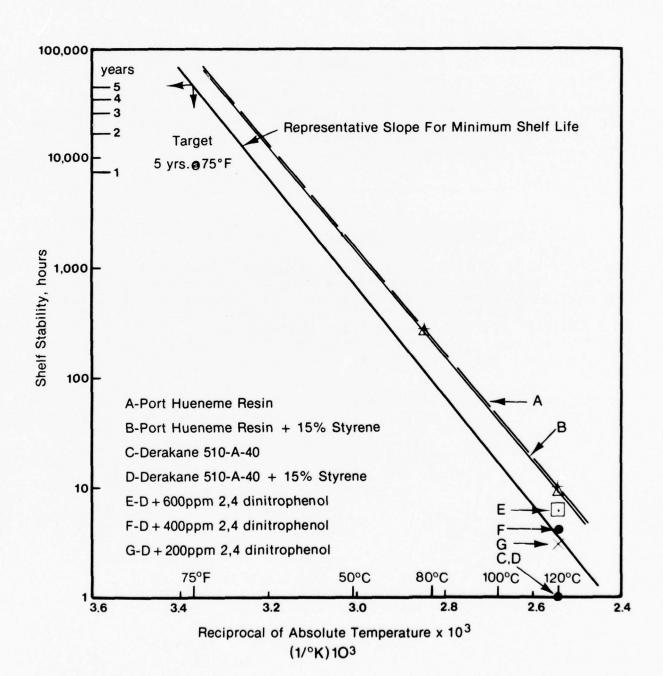
Stabilizer		Shelf Life, 120°C
0		Less than 1 hr
600 ppm THQ		2 hrs
600 ppm DNP		6 hrs
400 ppm DNP		4 hrs
200 ppm DNP		2 hrs
400 ppm THQ+200	ppm DNP	5 hrs
300 ppm THQ+200	ppm THQ	5 hrs

<sup>2</sup>Kirtland AFB, "Expedient Surfacing Materials", by H. E. Filter and S. S. Drake, AFWL-TR-73-70, (Oct. 1973, The Dow Chemical Co.).

<sup>3</sup>E. W. Lard, R. G. Rice, E. E. Stahly, "Storage Stability of Unsaturated Polyesters", Ind. Eng. Chem. Prod. Res. Develop., Vol. 10, No. 4, P. 391 (1971).

Figure 16

# SHELF LIFE vs TEMPERATURE



The addition of 600 ppm of 2,4 dinitrophenol does result in significant stabilization increasing the 75°F shelf life to 5 years. Unfortunately, this also results in decreased reactivity and a satisfactory cure can no longer be obtained with benzoyl peroxide or cumene hydroperoxide with the resin containing 400 ppm or 600 ppm of 2,4 dinitrophenol. The toluhydroquinone and 2,4 dinitrophenol did not show any synergistic stabilization with the DERAKANE 510-A-40 plus 15 percent styrene as was observed by Drake, et al<sup>1</sup>, for the Port Hueneme resin.

## SECTION VII

#### CONCLUSIONS

A system of chemical components has been developed for the field placement of fiberglass reinforced plastic soil surfacings under wet conditions that is superior to that presently used and is usable with the basic spray equipment now used by the Marine Corps. It does require an additional spray and rolling step before application of the resin.

The presence of 70 percent water in the FABMAT C-4020 and in the soil results in laminates that have flexural strengths of about 4,000 psi when fabricated from the presently used Selectron RS 50338 resin. The same flexural strength is obtained whether the wet fiberglass mat is used on a dry or wet substrate. The presence of as little as five percent moisture in the mats results in a 75 percent loss in flexural strength. This loss in strength is due to the lack of resin penetration into the woven roving layer of the FABMAT C-4020. Laminates prepared from chopped strand fiberglass mats containing 25 percent water that permit easy resin penetration, have a negligible loss in flexural strength as compared to a dry mat prepared laminate.

The addition of styrene to the Port Hueneme resin (RS50338) results in decreased viscosity and improved flexural strengths. It was found that the optimum results were obtained on the addition of 15 percent of styrene. At this styrene concentration the lowered resin viscosity, 80 cps, resulted in a flexural strength of 18,770 psi for a laminate prepared from water saturated FABMAT C-4020, which is a considerable improvement over the 4,000 psi obtained with undiluted Port Hueneme resin.

The incorporation of a nonionic surfactant, Triton X100, into the water in the fiberglass mat results in better resin penetration, or better water displacement, and increased flexural strength. The use of 1 percent solution of Triton X100 in the FABMAT C-4020 results in a flexural strength of 17,000 psi using laminates prepared from Port Hueneme resin and saturated mats. Under the same conditions, saturated mats, but using Port Hueneme resin plus 15 percent styrene, flexural strengths of 25,000 psi were obtained. This is within 10 percent of the desired 28,000 psi. The addition of the styrene does not change the shelf life of the resin as measured by gelation.

In field operations under wet conditions the real shelf life will be greatly reduced since these flexural strengths can not be obtained with high resin viscosity.

At 120°F the desired flexural strength can be obtained under wet conditions without surfactant or resin dilution. At 33°F flexural strengths greater than 10,000 psi were not obtained with either the use of a surfactant or diluted Port Hueneme resin.

DERAKANE 510-A-40, a brominated vinyl ester resin, when diluted with 15 percent styrene gives a flexural strength of 24,000 psi under wet conditions. When the saturated FABMAT C-4020 contains 1 percent Triton X100, laminates can be produced having flexural strengths of 30,000 psi; meets specification. This resin does not have the desired shelf life or reactivity.

All of the laminates prepared from saturated mats using Port Hueneme resin, styrene diluted Port Hueneme resin and DERAKANE give tensile strengths greater than 16,000 psi, well over the specified 13,000 psi.

Efforts to improve the flexural strength of laminates prepared under wet conditions by silane treatment of the fiberglass mat was not effective.

#### SECTION VIII

# RECOMMENDATIONS

A chemical component system has been developed that can be used to construct fiberglass reinforced plastic surfacing for soils under wet conditions. It is recommended that a field trial be made. This should be done to determine if field fabrication will result in the development of deficiencies in the incorporation of the surfactant and resin into the wet fiberglass mats. These tests should be made using both Port Hueneme resin (RS50338) plus 15 percent styrene and DERAKANE 510-A-40 with 15 percent styrene.

Development costs prohibited complete investigation in several areas. In view of this it is recommended that further work be done on the following subjects:

- 1. Studies should be made to determine the optimum surfactant and method of application to incorporate the surfactant into the water in the fiberglass mats.
- 2. A stabilizer should be developed to increase the shelf life of DERAKANE 510-A-40.
- 3. A need exists for a more active and controllable catalyst for stabilized DERAKANE 510-A-40 plus styrene.
- 4. Investigate other vinyl ester resins.

It is recommended that further efforts be made to obtain a woven fiberglass fabric mat made from spun strand roving with a chromium size to determine if the improved resin penetration into such a fabric would permit the use of more viscous resins.

SECTION IX

APPENDIX
TEST RESULTS

TABLE 1

RESIN REACTIVITY

(All tests made using 4% Vanadium Ten Cem, Control S5335, except where noted)

Barcol <sup>5</sup> Hardness		47/48	47/46	Cured	30/47	30/42	45/44	44/46	45/45	48/44	48/44	43/40	45/38	37-43/42
Min		20	9 2		t two runs.	56	15	24	50	12	50	80	1	15
Reactivity		152	176	133	sh for next	70	145	133	153	148	153	176	163	149
Read Gel Time Min		11.8	6.1	17.6	n in di	0.6	7.9	12.0	10.6	7.1	11.0	5.6	6.9	8.8
DDM <sup>4</sup>		00	00	0	grams of resi	0	0	0	0	0	0	0	0.1	0.2
DMT <sup>3</sup>	<b>ω</b> Ι	0.10	0.124	0.076	the 20	0.10	0.15	0.10	0.12	0.15	0.10	0.15	0.15	0.15
$\frac{\text{yst - phr}}{\sqrt{2}}$	RS 5033	0.15	0.186	0.114	added to	0.15	0.1	0.1	0.1	0.15	0.15	0.225	0.225	0.225
Catalyst CHP <sup>1</sup>	Port Hueneme Resin RS 50338	0.75	1.25	0.75	0	1.0			1.0	1.0	1.0	1.5	1.5	1.5
Test No.	Port Huer	0824A 0824B	0824C	0825A	20 grams 08240	0824F No Water	0116A	01168	01160	01160	0116E	1013A	10138	10130

(Continued)

TABLE 1 (Continues)

Ľ	Barcol Hardness	47/47	46/42	47/41-46	Cured	33-45/44	43-46/44	45-48/42	38-45/43	43/43	44/43	46/45	46/42-47	43/43	45/43-46	40-45/42	42-45/43		45-48/40-45	43-47/39-43	46/41	42/33-38	45/35-38		
	Min	24	43	12	19	31	99	9	7	œ	12	56	192	12	28	10	15		91	50	40	44	12		
Reactivity	Exotherm A Toc	140	146	146	148	135	122	185	186	202	182	168	165	159	138	167	153		149	140	143	109	165		
Rea	Gel Time Min	12.4	28.3	7.2	11.3	29.3	61.4	3.8	4.6	5.3	8.4	20.1	183	7.2	17.2	7.2	10.2		8.2	10.0	15.8	15.2	8.1	(pani	,
	DDM <sup>4</sup>	0.225	0.3	0.2	0.3	0.4	0.5	0	0.1	0.2	0.3	0.4	0.78	0.5	9.0	9.0	0.64		0	0	0	0	0.2	(Continued)	
	DMT <sup>3</sup>	0.15	0.15	0.18	0.18	0.18	0.18	0.2	0.2	0.2	0.2	0.2	0.2	0.25	0.25	0.3	0.3	tyrene	0.10	0.10	0.075	0.07	0.15		
talyst - phr	ν <sup>2</sup>	0.225	0.225	0.27	0.27	0.27	0.27	0.3	0.3	0.3	0.3	0.3	0.3	0.375	0.375	0.45	0.45	Resin + 10% Styrene	0.15	0.15	0.1125	0.105	0.225		
Catalys	CHP	1.5	1.5	1.5	1.5	1.5	1.5	2.0	2.0	2.0	2.0	2.0	2.0	2.5	2.5	2.5	2.5		•			0.56	•		
	Test No.	1012	1013E	1017A	1014A	10178	10170	09138	0913A	09130	09148	0914C	0914A	1215A	12158	12150	12150	Port Hueneme	1209A	0323A	0323B	1209B	12090		

TABLE 1 (Continued)

Barco15	Hardness	42/38 45/44 40/37 41/36 43/39
	Min	18 11 11 31 31
eactivity me Exotherm	D OIO	151 146 160 164 160
Gel Tin	Min	9.3 7.9 6.5 9.0
	DDM <sup>4</sup>	0.3 0.4 0.5 0.55 0.625
	DMT <sup>3</sup>	0.15 0.15 0.25 0.25 0.25
t - phr	٧٢	0.225 0.225 0.3 0.375 0.375
Catalys	문	2.5 2.5 2.5 2.5 2.5
	No.	1212A 1212B 1212D 1213B 1213C

(Continued)

TABLE 1 (Continued)

Barcol <sup>5</sup> Hardness		41/36	46/39	45/37		46/37	5)40 Shattered Shattered		47/44	3//23	45/40	43/33-38	Cured	Cured	Shattered	Shattered	Shattered	Shattered		Cured						
Min		23		25		25	39 14 22		52	89	22	51	8 %	38	. 6	15	17	35 25	3	71				hthalate		
Reactivity ime Exotherm		162	167	146		177	202 199		127	:	135	92	144	30	179	191	164	163	2	137				dimethylu		
Reac Gel Time Min		10.9	8.7	14.0		10.5	9.4 1.5		11.3	21.5	10.5	20.2	8 F	7.6	7.0	11.2	13.2	14.3		7.9				Deroxide in		
P <sub>M</sub> QQ		0	0 45	0.5		00	0.45 0.5	43	0 (	0	0	0	00	<b>-</b>	0.34	0.406	0.438	0.438	?	0				PERSOL-DDM, 60% methylethyl ketone peroxide in dimethylphthalate		
DMT <sup>3</sup>	Styrene	0.10	0.15	0.25	yrene	0.10	0.25	Resin V-Control LB-267-43	0.10	0.075	0.10	0.075	0.10	0.0/2	0.25	0.25	0.25	0.25	3	0.10	ride		coluidine	methylet		
$v^2$	+ 15%	0.15	0.225	0.375	1 + 20% Styrene	0.15	0.375	V-Contro	0.15	0.1125 ter	0.15	0.1125	0.15	0.1175	0.375	0.375	0.375	0.375	separately	0.15	nene hydroperoxide	TEN CEM	N dimethyl-p-toluidine	DDM, 60%	- Ton/Rottom	
Catalyst CHP <sup>l</sup>	Hueneme Resin		•	2.5	neme Resin	•	2.5		1.0	0./5 batch promoter	_	0.75	•	•		•		2.5	ed :	0.	Cumene	4% V TE	N, N dir	IN I	Handness	222
Test No.	ىد ا	0127A	01278	01270	Port Hueneme	0215A	02150 02150 0215C	Port Hueneme	0227A	0227B Fresh ba		0227E	03010	0301E	0228F	0228D	0228B	0228C	Vand DMT	0303A	- GHD	- Λ <sub>2</sub>	3 <sub>DMT</sub> -	4 DDM -	Sarcol Hardr	מוכסו

TABLE 2

INFLUENCE OF TEMPERATURE ON REACTIVITY

				React	tivity			
Temp Catalyst		t Composit		Gel Time Min.	Exoth	erm ∆T-°C	Barcol Hardness	
	Hueneme id Jacket	Resin + 15	% Styrene					
32 32 120 120 120 120	2.0 3.125 0.375 0.5 1.0	0.5 0.78 0.094 0.125 0.25	- - - 0.3125 0.375	18.5 14.2 10.3 6.4 8.4 13.3	46.2 37 - 10.5 17	92 63 - 127 136	35-40/33 39-40/0 21/0 42/34-38 Shattered Shattered	
Air 32 32	Jacket 2.0 3.125	0.5 0.78	:	12.6 9.1	67 47	30 73	39-40/36-38 39-42/0?	
	Hueneme id Jacket							
32 32 120 120	1.6 2.24 0.5 0.375	0.4 0.56 0.125 0.094	:	13.8 8.0 6.0 11.0	45.5 22. -	97 124 - -	42/15 42/17 38-42/42 35/38-42	

<sup>&</sup>lt;sup>1</sup>CHP - Cumene Hydroperoxide

 $<sup>^2</sup>$ Promoter - 3 parts 4% V Ten Cem $^{(R)}$  to 2 part N,N dimethyl-p-toluidine

 $<sup>^{3}</sup>$ \*DDM - LUPERSOL (R)DDM, 60% methylethyl ketone peroxide in dimethylphthalate

<sup>&</sup>lt;sup>4</sup>Barcol Hardness - Top/Bottom

TABLE 3

INFLUENCE OF PROMOTER AND INHIBITOR ON THE REACTIVITY

OF PORT HUENEME RESIN

	Cataly	st Compos	sition-ph	ir	R	eactivit	y	
Test No.	CHP1	<u>v</u> <sup>2</sup>	DMT <sup>3</sup>	DDM <sup>4</sup>	Gel Ti Min.	me Exot ΔT°C	herm <u>Min.</u>	Barcol <sup>5</sup> Hardness
0221E 0221F 0222B 0330A 0330B 0330D 0330G 0330E 0330F 0330C 0330H 0221H 0329C 0329B 0222D 0222F 0222E 0222C 0330I 0330J	1.0 0.75 1.0 1.0 1.0 1.0 1.0 1.0 2.5 2.5 2.5 2.5 2.5 2.5	0.15 0.1125 0.15 0.125 0.1 0.18 0.15 0.15 0.12 0.12 0.375 0.375 0.375 0.375 0.375 0.375	0.15 0.1125 0.15 0.125 0.1 0.12 0.12 0.10 0.10 0.08 0.375 0.375 0.375 0.375 0.375 0.375	- - - - - - 0.5 0.45 0.406 0.406 0.375 0.33 0.2	7.6 14.0 7.8 10.3 12.4 7.9 7.8 8.7 8.6 12.2 11.3 28 25.7 17.8 16.3 7.9 4.9 3.0 18.5 21.5	147 131 145 158 124 139 139 154 152 115 135 173 173 177 178 176 188 186	14 30.7 15.5 19 30 14.8 14.0 16.0 17.5 29 24.3 29.7 29 21 16.8 9.5 6.0 40	44-46/43-44 45-46/40 - 45-47/41-42 49/44-45 47-48/37 43-45/37-38 46-48/42 45-47/38-41 45-48/41-42 42-43/40-42 Shattered

<sup>&</sup>lt;sup>1</sup>CHP - Cumene hydroperoxide

<sup>&</sup>lt;sup>2</sup>V - 4% V TEN CEM (new batch) LB-267-43

 $<sup>^3\</sup>mathrm{DMT}$  - N,N dimethyl-p-toluidine

 $<sup>^4\</sup>mathrm{DDM}$  - LUPERSOL-DDM, 60% methylethyl ketone peroxide in dimethylphthalate

<sup>&</sup>lt;sup>5</sup>Rarcol hardness - Top/Bottom

TABLE 4

REACTIVITY OF SELECTRON SR 3703\*

	Catalys	t Compositi		Reac	ctivity	Barcol <sup>4</sup>
No.	CHP 1	Promoter <sup>2</sup>	DDM <sup>3</sup>	Gel Time	ΔT°C, Min.	Hardness
1111A 1111B 1111C 1111D 1111E 1111F 1111G 1111H 1114D 1115A 1115B 1115D 1116C 1121A 1121B 1121C	1.0 0.5 0.5 0.5 0.5 0.5 0.5 0.5 0.5 1.0	0.25 0.125 0.0625 0.25 0.25 0.125 0.125 0.125 0.25 0.25 0.25 0.25 0.25 0.25 0.25	0 0 0.4 0.2 0.3 0.2 0.1 0.25 0.25 0.25 0.25 0.21 0.2	<1.3 min 2.9 8.4 >32 5.2 26.5 - 10 15.9 17.5 16.9 17.1 8.7 5.1 29.6 2.4	175 - 2.5 177 - 4.8 113 - 16.25 163 - 78 153 - 9.5 159 - 30 184 - 61 Lost - 15 155 - 24 163 - 29 154 - 25.5 171 - 23 169 - 13.5 174 - 8 168 - 35 180 - 4.25	48-49/42-43 46-49/43-46 43-46/38-39 46-48/42-48 46-49/41-42 44/39-41 45-48/45-48 45-58/43-45 47/46-48 Cracked 45-47/42-43 46-47/43-44 45-47/42-43
11210	1.0	0.5	0.5	7.7	151 - 11.3	43-44/42-43

<sup>\*</sup> Same as Port Hueneme resin except no added inhibitor.

Cumene hydroperoxide.

<sup>2. 3</sup> parts 4% Vanadium TEN CEM, 2 parts N,N dimethyl-p-toluidine.

<sup>3.</sup> Lupersol  ${\rm DDM}^{(R)}$  - Methylethylketone peroxide in dimethyl phthalate.

<sup>4.</sup> Top/Bottom

TABLE 5

REACTIVITY OF KIRTLAND PRIMARY RESIN\*

	Cataly	st Composition	n, phr	React	tivity	Barcol <sup>4</sup>	
No.	CHP 1	Promoter <sup>2</sup>	DDM3	Gel Time	ΔT°C, Min	Hardness	
1116B	1.0	0.25	0.2	78 min	155 - 100	42/45/36-38	
1116D	1.0	0.25	0	4.1	175 - 8.5	43-46/44-45	
1116E	1.0	0.25	0.1	9.2	106 - 11	42-43/42	

- \* Kirtland Primary Resin 76% ALTEK<sup>(R)</sup> 8-52 and 24% DERAKANE 510
- 1. Cumene hydroperoxide.
- 2. 3 parts 4% Vanadium TEN CEM<sup>(R)</sup>, 2 parts N,N dimethyl-p-toluidine.
- 3. Lupersol DDM(R)- Methylethylketone peroxide in dimethylphthalate.
- 4. Top/Bottom

TABLE 6 REACTIVITY OF DERAKANE 510-A-40

	Catalyst	Compositio	n – phr	Reac	tivity	- Barcol <sup>4</sup>		
Test No.	CHP 1	Promoter <sup>2</sup>	DDM <sup>3</sup>	Gel Time	∆T°C Min	<u>Hardness</u>		
1207B	1.0	0.25	-	12.1 min	13-36	0		
1207C	1.0	0.5	-	9.2	85-22.5	36-38/27		
1208A	1.5	0.75	-	9.6	88-20	35-36/30-32		
1207D	2.0	0.5	-	-	56-22.5	32-34/16-17		
1208B	2.0	1.0	-	8.6	82-15.2	33-35/28		
	CoN <sup>5</sup>	DMA 6						
1208C	0.2	0.1	1.5	15.7	56-37.2	33-36/22		
1208D	0.2	0.2	1.5	11.2	82-21.8	37-38/27-29		

<sup>1 -</sup> Cumene hydroperoxide.
2 - 3 part 4% Vanadium Ten Cem, 2 parts N,N dimethyl-p-toluidine
3 - Lupersol DDM - Methylethylketone peroxide in dimethylphthalate.
4 - Top/Bottom
5 - Cobalt napthenate, 6% Co
6 - N,N-dimethylaniline

TABLE 7

REACTIVITY OF DERAKANE 510-A-40

WITH 15% ADDITIONAL STYRENE

Benzoyl Peroxide Catalyst

				activit	у	
Test No.	Catalyst BZQ-40 <sup>1</sup>	- phr DMA <sup>2</sup>	Gel Time <u>Min.</u>	Exoth <u>\Delta T^C</u>	erm <u>Min.</u>	Barcol Hardness
0126D	2.66	0.21	15	83	29	33/25
0126E	3.75	0.3	9.2	92	20	30/22

 $<sup>^{1}</sup>$ BZQ-40; 40% Benzoyl peroxide pourable paste (Witco Chemical Corporation)

 $<sup>^2\</sup>mathrm{DMA}$  - N,N Dimethylaniline

TABILE 8 REACTIVITY OF DERAKANE 510-A-40 CONTAINING 15% STYRENE

Test No.	Catalys CHP <sup>1</sup>	$\frac{t - ph}{v^2}$	DMT <sup>3</sup>	Gel Time <u>Min.</u>	Exothe <u>AT°C</u>	erm <u>Min.</u>	Barcol <sup>4</sup> Hardness
1221A 1221C 1221B 0123C Resin + 0310A 0310B	1.0 1.0 2.0 2.5 600 ppm 2.0 3.0	0.3 0.6 0.6 0.45 2,4 0.6 0.9	0.2 0.4 0.4 0.3 dinitrop 0.4 0.6	19.3 23.5 16.2 20.7 heno1 45 33.3	45 - 66 - 4	59 >60 36 >55 - ~65	21/6 - 33/22 - 0 0
1215E 1215F 1227A 1227B 1227C 1227D	DDM <sup>3</sup> 1.0 2.0 1.0 1.5 1.0	0.15 0.3 0.15 0.225 0.2	CoN / 0.15 0.3 0.3 0.45 0.4 0.6	24.5 17.6 15.7 11.3 13.0 10.0	<10 42 35 63 60 78	>62 42 51 32 36 21	0 28/2 18/0 30/21 32/15 35/20

<sup>Cumene hydroperoxide
4% Vanadium Ten Cem
N,N dimethyl-p-toluidine
Top/Bottom
Lupersol DDM - methyl ethyl ketone
N,N - dimethylaniline
Cobalt napthenate, 6% Co</sup> 2 -3 -4 -5 -6 -7 -

TABLE 9
EFFECT OF WATER ON FLEXURAL STRENGTH

Port Hu	eneme Re	sin						
Com- posite No.	Water%	Catal	yst - ph Pro- moter <sup>2</sup>	DDM <sup>3</sup>	Gel Time Minutes Surplus	Exo- therm <u>Min.</u>	Resin	Flexural Strength psi
0912	0	0.96	0.24	0		29		39,600
1025A	4.8	1.0	0.25	0	11	20	58.9	10,830
1024A	10.2	1.0	0.25	0	8	16	59.0	12,030
0928A	25.8	2.0	0.5	0.4	-	20	58.7	12,960
0928B	25.8	2.0	0.5	0.4		28	50.2	11,700
1012A	27.4	1.5	0.375	0.225	13	27	61.9	13,460
1011B	27.8	2.0	0.5	0.31	8	14	63.0	7,280
1007W	28.4	2.0	0.5	0.31	-	28	59.9	17,210
1027A	50.4	2.0	0.5	0.32	11	34	61.9	6,140
1018B	71.4	1.5	0.45	0.3	11	34	63.0	3,620
1018C	71.7	2.0	0.5	0.31	8	23	60.4	3,860

<sup>&</sup>lt;sup>1</sup>CHP - Cumene Hydroperoxide

<sup>&</sup>lt;sup>2</sup>Promoter - 3 parts 4% V Ten Cem<sup>(R)</sup> to 2 part N,N dimethyl-p-toluidine

 $<sup>^3 \</sup>text{DDM} - \text{LUPERSOL}^{\text{(R)}}$  DDM, 60% methylethyl ketone peroxide in dimethylphthalate

TARIF 10

COMPOSITES PREPARED ON WATER SATURATED SAND WITH PORT HUENEME RESIN

	loven up, not sequential	=	=	loven up, sequential	=	=	Woven down, sequential
S)	nb,	=	=	nb,	=	=	down
Remarks	Woven	=	•	Woven	=	= **	Woven
Flexural Strength psi	4,630	8,600	4,450	09,760	14,680	16,600	14,400
Resin %	59.3	62.4	63.1	55.3	59.1	61.5	63.1
Exo- therm Minutes	18	16	31	32	28	28	22
Gel Time Minutes Surplus	10	80	10	1	13	14	12
DDM <sup>3</sup>		0.31	0.31	0.225	0.225	0.225	0.225
yst - phr Promoter <sup>2</sup>	0.25	0.5	0.5	Ĭ	0.375	0.375	0.375
Catal CHP 1	1.0	2.0	2.0	1.5	1.5	1.5	1.5
Mat Water %	0	0	70.4	0	0	0	0
Com- posite No.	10278	1028A	10288	1101A	1103A	1103B	1110A

\*\*Dichlorodimethyl silane treated mat - washed

1CHP - Cumene Hydroperoxide

 $^2$ Promoter - 3 parts 4% V Ten Cem $^{(R)}$  to 2 part N,N dimethyl-p-toluidine

 $^3 ext{DDM}$  - LUPERSOL $^{(R)} ext{DDM}$ , 60% methylethyl ketone peroxide in dimethylphthalate

TABLE 11
INFLUENCE OF FABRICATION METHODS ON FLEXURAL STRENGTH\*
All tests Port Hueneme resin RS50338

Remarks	Woven up, sequential	Woven up, sequential	Woven up, not sequential	Woven down, not sequential	Woven down, not sequential	Woven down, sequential,	Woven down, sequential,	resin poured not rolled Woven down, sequential	Woven down, not sequential	One layer Fabmat C-4020	One layer Fabmat C-4020
Flexural Strength psi	11,050	16,300	13,460	19,260	17,600	14,110	13,060	35,990	39,600	1,860	13,660
Resin %	61.3	60.4	6.19	8.09	63.4	66.2	1.99	65.4	1	61.4	64.9
Exotherm Minutes	27	:	27	28	12	21	22	19	53		23
Gel Time Minutes Surplus	Ξ	6	13	12	80	12	13	10	1	Ξ	12
DDM <sup>3</sup>	0.21	0.225	0.225	0.2	0.31	0.21	0.21	0	0	0.3	0.21
/st - phr Promoter <sup>2</sup>	0.375	0.375	0.375	0.375	0.5	0.375	0.375	0.25	0.24	0.45	0.375
Cataly	1.5	1.5	1.5	1.5	1.75	1.5	1.5	1.0	96.0	1.5	1.5
Mater Water %	25	27	27.4	26.4	25.8	25.7	27.0	0	0	70.6	5.2
Composite	11048	10238	1012A	11078	11070	1109A	11098	1108A	0912	1019A	1109E

(Continued)

TABLE 11 (Continued)

	Remarks	One layer Fabmat C-4020	One layer Fabmat C-4020	Six layers of 2 oz./ft <sup>2</sup>	Six layers of 2 oz/ft <sup>2</sup>	Six layers of 2 oz/ft	Chopped Strand mat Six layers of 2 oz/ft <sup>2</sup> chopped strand mat	Four layers of 18.5 oz.	layers 2 oz./ft <sup>2</sup> of chopped strand
Flexural	psi	23,500	22,320	21,750	18,360	19,530	14,440	41,000	9,830
Bosin	8	60.3	58.4	61.8	63.2	62.4	63.2	58.0	56.4
F < 0 + be can	Minutes	36	34	56	32	50	15	28	27
Gel Time Minutes	Surplus	10	=	∞	10	9	6	∞	80
	DDM <sup>3</sup>	0	0.21	0	0.21	0.32	0.55	0	0.31
yst - phr	Promoter <sup>2</sup>	0.25	1.5 0.375	0.25	0.375	0.5	0.625	0.25	0.5
Catal	CHP	1.0	1.5	1.0	1.5	2.0	2.5	1.0	2.0
Mat	2%	0	0	0	25.5	26.0	8.69	0	24.5
Com-	No.	1020A	10200	1206A	12068	1207A	0109A	1214C	12140

\*All composites 2 layers of FABMAT C-4020 except where noted.

 $^2 Promoter$  - 3 parts 4% V Ten Cem $^{\rm (R)}$  to 2 part N,N dimethyl-p-toluidine  $^3 DDM$  - LUPERSOL  $^{\rm (R)} DDM$ , 60% methylethyl ketone peroxide in dimethylphthalate CHP - Cumene Hydroperoxide

TABLE 12
INFLUENCE OF RESIN VISCOSITY AND SURFACTANTS ON FLEXURAL STRENGTH

Remarks	ביין די יין ניין אר נייד	X - % solution  riton		TX.5-0.5% solution Triton	CF-1% solution Triton	W-% Water in Mat then sprayed with 1% sol'n	סן ורונטון אוטט			P-0.5% sol'n of Pyronate 50	A/c - 2 B alcohol (ethyl	alconol)	
Flexural Strength	000	25,280	17,670	16,820	57.9 22.860	16,590	14,520	14,610	16,740		21,490	20.770	
Resin %		59.9	58.4	57.5	57.9	55.5	56.8	9.99	60.3		58.0	59.7	
Exo- therm Resin Min. %	8	97	37	52	41	33	1	27	32	Did not cure	56	37	
Gel Time Minutes Surplus		,	12	80	13	10	12	80	10	Did n	7	œ	(Continued)
hr DDM <sup>3</sup>		0.55	0.55	0.46	0.55	0.55	0.55	0.45	0.45	0.47	0.55	0.55	0)
Catalyst - phr Promo- CHP <sup>1</sup> ter <sup>2</sup> DI		629.0 6.2	0.625	0.625	2.5 0.625	2.5 0.625	2.5 0.625	2.5 0.625	0.625	0.625	2.5 0.625	2.5 0.625	
Cata CHP		7.5	2.5	2.5	2.5	2.5	2.5	2.5	2.5	2.5	2.5	2.5	
Mat Fluid %	12.70	1716.62	69.6TX1 2.5 0.625	72.9TX.5 2.5 0.625	69.1CF	25.1W 5.1TXI	24.8W	71.4W	69.0W	72.4P	24.7W	24.4	10. IA/C
Resin		UIISA Port Hueneme 25.51XI								-	-		
Com- pos- ite No.	40110	01184	0123A	03130	0214C	0117A	01178	0223A	02238	0309A	01188	0119A	

								led						
	Remarks							Paddle rolled						
	Flexural Resin Strength % psi	60.7 45,160	23,430	20,850	23,080	12,440	24,610	23,450	58.0 42,260	18,770	23,320	26,850	25,800	
	Resin %	60.7	65.0	61.0	59.7	9.09	55.7	61.7	58.0	61.0	57.2	57.6	9.99	
	Exo- therm Min.	11	12	23	56	44	:	18	19	38	34	23	30	
	Gel Time Minutes Surplus	15	10	∞	6	12	;	7	Ξ	6	10	7	11	(Continued)
	phr DDM <sup>3</sup>	0.3	0.55	0.55	0.55	0.55	0.55	0.55	0	0.47	0.48	0.4	0.46	(Cont
	lyst - Promo- ter <sup>2</sup>	1.5 0.375	0.625	0.625	0.625	0.625	0.625	0.625	1.0 0.25	0.625	0.625	0.625	2.5 0.625 0.46	
	Cata	1.5	2.5	2.5	2.5	2.5	2.5	2.5	1.0	2.5	2.5	2.5	2.5	
ed)	Mat Fluid %	O •	24.9W	25.5W	24.1TX1	70.8W	68.1TX1	24.9W	0	W6.07	70.4TX1	70.2TX1	73.4CF	
TABLE 12 (Continued)	Resin	1219A Port Hueneme + 10% Styrene	=			=	=		0201B P.H. + 15% Styrene	=			=	
TABLE	Com- pos- ite No.	1219A	12198	12190	01248	1220A	0124A	02148	02018	0201A	0213A	0301A	0301F	

TABLE 12 (Continued)

Remarks	Wet mat sprayed with 10% sol'n of Triton X100	Wet mat sprayed with 2B alcohol							1.5 phr DDM, 0.2 phr DMA, 0.2 phr CoN	1.5 phr DDM, 0.2 phr DMA,	ore pile coll
Flexural Resin Strength	19,660	10,360	44,420	21,750	12,340	40,520	15,240	14,310	42,100	3,620	
Resin %	59.2	58.1	56.8	56.4	54.2	56.8	9.09	65.0	59.4	8.09	
Exo- therm Min.	1	1	52	31	32	17	15	18	56	!	
Gel Tíme Mínutes Surplus	6	12	12	10	14	7	6	7	12	12	(Continued)
phr DDM <sup>3</sup>	0.5	0.47	0	0.47	0.47	0	0	0	1	1	(Con
Catalyst - phr Promo- CHP <sup>1</sup> ter <sup>2</sup> DDM	0.625	2.5 0.625 0.47	0.25	0.625	0.625	1.0	1.0	1.0	1	1	
Cata CHP	2.5		1.0	2.5	2.5	2.0	2.0	2.0	:	1	
Mat Fluid %	W0.69	70.3W 13.2A/c	0	26.0	70.3	0	24.6W	70.9W	0	24.7W	
Resin	0213B P.H. + 15% Styrene	=	0216B P.H. + 20% Styrene		s	0111B Derakane 510-A-40					
Compos- ite No.	02138	0302F	02168	0216A	0215B	01118	01108	01198	ALLTO	0110A	

TABLE 12 (Continued)

CHP - Cumene Hydroperoxide 2Promoter - 3 parts 4% V Ten Cem<sup>(R)</sup> to 2 part N,N dimethyl-p-toluidine 3DDM - LUPERSOL<sup>(R)</sup> DDM, 60% methylethyl ketone peroxide in dimethylphthalate

TABLE 13
MISCELLANEOUS RESIN LAMINATES

Remarks				dichlorodimethyl silane	רו במרבת ווומר		On 100% Saturated Sand		On Dry Sand
Flexural Strength	30,740	20,190	13,870	10,600		17,850	25,590		36,770 36,710
Resin %	60.3	58.3	64.2	64.9		60.7	61.7		61.0
Exo- therm Min.	20	8 6	2	10		50	22		12
Gel Time Minutes Surplus	14	==	9 0	10		თ	9		ഗ ശ
ohr DDM <sup>3</sup>	0.25	0.25	0.5	0.55		0.1	0.11		00
Catalyst - phr Promo- CHP <sup>1</sup> ter <sup>2</sup> D	0.25	0.25	0.5	0.5		0.25	0.25		1.27 0.318 1.27 0.318
Cata CHP	0.5	0.5	.0.	1.0		1.0	0.1		1.27
Mat Fluid	0	5.2W	25.3W	24.7W		29.6M	0		00
Resin	1115E Selectron SR3703		=	=	1122A Kirtland	Primary		Port	Hueneme "
Com- pos- ite No.	1115E	1114A	1121F	11288	1122A		1122B	1123A Port	11238

1CHP - Cumene Hydroperoxide

 $^2$ Promoter - 3 parts 4% V Ten Cem $^{(R)}$  to 2 part N,N dimethyl-p-toluidine

 $^3 \mathrm{DDM}$  - LUPERSOL $^{(\mathrm{R})} \mathrm{DDM}$ , 60% methylethyl ketone peroxide in dimethylphthalate

EFFECT OF TEMPERATURE ON FLEXURAL STRENGTH TABLE 14

			Mat	Catal	Catalyst - phr			
Composite No.	Resin	Temp ° F	Fluid %	H	Promoter <sup>2</sup>	DDM <sup>3</sup>	Resin %	Flexural Strength psi
0321A	Port Hueneme	33	74.1TX1 0	2.0	0.5	00	58.5	4,290 35,400
0350A	Styrene	33	72.3TX1	3.0	0.75	0	56.3	9,240
;	Port Hueneme	120	0	0.5	0.125	0	1	42,800
0327A 0322A 03235	Port Hueneme Port Hueneme	220	73.4W 70.2TX1	0.0	0.25	0.3	61.8 60.1	33,990 33,410
13250	Styrene	120	1X1.017	1.0	0.25	0.33	59.1	35,470

1CHP - Cumene Hydroperoxide

<sup>2</sup>Promoter - 3 parts 4% V Ten Cem<sup>(R)</sup> to 2 part N,N dimethyl-p-toluidine

<sup>3</sup>DDM - LUPERSOL<sup>(R)</sup>DDM, 60% methylethyl ketone peroxide in dimethylphthalate

TXI - 1% Triton X100 W - Water

TABLE 15 LAMINATE TENSILE STRENGTH

		75°F Ambient Temperature							
	Remarks	75°F Ambie							
Tensile	Strength psi	17,240	18,050	20,500	18,800	16,630	23,350	22,700	26,300
	Resin %	61.3	62.4	1	57.7	61.2	9.19	62.0	9.19
	therm Min.	34	33	;	43	1	19	44	23
Gel Time	Minutes Surplus	10	10	;	=	10	12	19	16
hr	DDM <sup>3</sup>	0.47	0.47	0	0.5	0.5	0	0	0
Catalyst - phr	ter <sup>2</sup>	0.625 0.47	0.625 0.47	0.25	0.625	0.625 0.5	0.25	1.25	1.25
Catal	GRP.	2.5	2.5	1.0	2.5	2.5	1.0	2.5	2.5
Mat	Fluid %	71.4	70.6TX1	0	70TX1	72.2W	0	70.5TX1 2.5	0
	Resin	0314A Port Hueneme 71.4	0314B Port Hueneme	Port Hueneme	0314C Port Hueneme 70TX1 + 15% Styrene	=	=	0316A Derakane 510- A-40 + 15% Styrene	:
Com-	ite No.	0314A	0314B	1	03140	0315A	03158	0316A	03168

2promoter - 3 parts 4% V Ten Cem<sup>(R)</sup> to 2 part N,N dimethyl-p-toluidine 3DDM - LUPERSOL(R) DDM, 60% methylethyl ketone peroxide in dimethylphthalate CHP - Cumene Hydroperoxide W - Water TX1 - 1% Triton X100

TABLE 16

INFLUENCE OF TEMPERATURE ON RESIN VISCOSITY

	Viscosity - cps			
Port Hueneme Resin	Port Hueneme Resin + 15% Styrene			
1050	260			
218	80			
63	28			
	1050 218			